# MIX-PROPORTION AND MIXING PROCEDURE FOR STABLE ENTRAINED AIR IN SELF-COMPACTING CONCRETE


BY

RATH SOVANN SATHYA



DISSERTATION

Submitted in partial fulfillment of the requirements for the degree of

Doctor of Engineering in Concrete Engineering,

Graduate School of Systems Engineering,

Kochi University of Technology,

Kochi, Japan


Review Committee:

Professor OUCHI Masahiro, Supervisor

Professor SHIMA Hiroshi, Co-supervisor

Professor KAI Yoshiro, Co-supervisor

Professor TAKAGI Masataka, Member

Professor NASU Seigo, Member

Associate Professor GOSO Takashi, Member


September, 2016

# ABSTRACT

The purpose of this study is to establish a method for entraining stable air with a target air volume. A controllable air entrainment is necessary for production of air-enhanced self-compacting concrete. Stable air entrainment can prevent errors in concrete strength design and ensures the self-compactability of fresh concrete with the time passed.

Experiments were conducted with fresh mortar to evaluate the stability of air in fresh stage resulted from different mixing procedure. Mortar was chosen for experiment instead of concrete since it is more flexible when various adjustments of mix-proportion and mixing procedure was attempted to be conducted. With mortar samples produced by different mixing procedure,  the critical size of bubbles to the stability of air was firstly determined using air distribution test results at fresh stage of mortar obtained from the air void analyzer (AVA-3000). To improve the stability of air, it is indispensable to minimize the air volume of coarse bubbles.

It was found that instability in volume of air in fresh mortar was caused by the existence air bubbles with chord length of mainly over 1 mm and partly of 0.5 mm to 1 mm due to unification between air bubbles. To ensure the stability of air, the critical size was chosen to be 0.5 mm to which the air volume of bubble larger than this size needed to be minimized. Different characteristic in entraining air entrainment of different mixing procedure was influenced by viscosity. Higher viscosity of fresh mortar during air entrainment in mixing resulted in higher efficiency in volume of air entrainment. The upper limit volume of fine entrainment was proportional to the dosage of air-entraining agent. When the upper limit of fine air was reached by the mixing time passed the coarse air volume start increasing.

# ACKNOWLEDGEMENT

This dissertation would not be successfully accomplished without the contribution of many people giving valuable supports including financials, academic advices and comments, encouragements, and beliefs.

I would like to firstly express my deepest gratitude to my supervisor, Prof. OUCHI Masahiro who has provided endless supports, precious ideas, and great comments. Without his supervision, this dissertation would not be successfully completed. I am also sincerely grateful to be advised with countlessly great recommendations from both of my co-supervisors, Prof. SHIMA Hiroshi and Prof. KAI Yoshiro. All those indispensable ideas enhanced the reliability of my research work. I am thankful to be continually supported and encouraged by Prof. TAKAGI Masataka, Prof. NASU Seigo and Associate Prof. GOSO Takashi. Without their enlightening advices, this dissertation would not be smoothly accomplished. I would like to express my sincere gratitude to Mr. MIYAJI Hideo, a technical instructor in our Concrete Laboratory, for facilitating my experimental works and for his warm support. Also, I am thankful for all the good collaborations coming from our laboratory's members. I am thankful for the special scholarship program (SSP) of the Kochi University of Technology for the financial supports with the warm hospitality from all IRC's staffs.

Another special thank is to my parents for always giving me the full supports and encouragements to fulfill my dream. Without their endless loves and compassion, I would not be who I am today. For more than two decades of raising me up, they put my education as priority regardless the hardship they have to bear with. Finally, I would like to express special thanks to the one who has been going through all the up and down moments together.

# TABLE OF CONTENTS

# CHAPTER 1 INTRODUCTION

## 1.1. Background

### 1.1.1. Self-compacting concrete

Self-compacting concrete (SCC) is defined as concrete having self-compactability, a capacity of concrete to be uniformly filled and compacted into every corner of formwork by its own weight without vibration [1]. SCC was proposed by *Okamura* (1986) to achieve durable concrete independently of the quality of construction workers. Fundamental study on the workability of SCC and its development were then conducted by *Okamura*, *Maekawa* and *Ozawa* [2-4]. The prototype of SCC, which was first completed in 1988, was then named as self-compacting high performance concrete to differentiate its performance from the other high performance concrete (HPC) [5]. To achieve self-compactability of self-compacting high performance concrete, *Okamura* and *Ozawa* have limited aggregate content, lowered water to powder ratio, and used superplasticizer [6]. Powder content includes cement content and content of fine materials such as fly ash, blast furnace slag, and other supplementary cementitious materials. Superplasticizer (SP) is a high-range water-reducing agent needed to produce flowable concrete. SP belongs to a group of chemical acting as dispersant preventing the flocculation of fine particles such as cement thus freeing pocket water and increasing the flowability of concrete accordingly.

Since the development in 1988, SCC has been used particularly in the structural elements with confined reinforcement or complicated formwork. Advantages for using SCC include self-compactability, environmental friendly application, and reduction in construction time. However, in spite of these advantages, application of SCC has been limited by its expensive cost. High powder content usage, which is one of the key factor to achieve self-compactability in conventional SCC, has become a hindrance to the application of SCC in general construction because of the high unit cost. To lower the unit cost of the conventional SCC, fly ash has been a well-known substitution material to replace some portions of cement content in the mix-proportion.

In this study, instead of using supplementary cementitious material, the author aimed to introduce higher entrained air bubbles content for higher water to cement ratio and fine aggregate content in mortar in the mix-proportion of SCC so that the unit cost can be reduced accordingly.

### 1.1.2. Conventional air entrainment to concrete

***a) Application of air entrainment***

Air entrainment to concrete was found beneficial to improve the freeze-thaw resistance of concrete exposed to a severe weather condition. Entraining air to a concrete has become no exception for concrete serving in the cold-weather country [7, 8]. Air content required to ensure the freeze-thaw resistance depends on the maximum size of the coarse aggregate and the condition of the weather. According to ACI 318 [9], for concrete produced with the nominal maximum size of coarse aggregate of 25cm, the air content required to achieve the freeze-thaw resistance was 6% and 4.5% for severe or very severe exposure and moderate exposure respectively. Additionally, other parameters of the air-void system such as the spacing factor and specific surface area of air are also important criteria for the freeze-thaw resistance. The spacing factor is the average distance between individual air bubbles in the matrix of concrete. The specific surface area of air is the average surface area of air per unit volume of air. The critical value for these parameters was varied. Usually the freezing and thawing test was conducted on concrete samples of different air-void system to determine the critical value and to clarify the influence of different factors on those parameters.

Air entrainment was also reported to reduce bleeding and increase plasticity of concrete [10]. Although, air entrainment was found improving the durability of concrete in hardened stage, the higher content of air reduces the strength of concrete. Generally, compressive strength of concrete was reduced around 5% for every percent of air content [10].

***b) Size of air bubbles in air entrainment***

There are two types of air in concrete: entrained air and entrapped air. Entrained air is intentional air incorporated either by air-entraining cement or air-entraining agent (AE). Entrapped air is accidentally created during mixing, consolidating or placement of concrete. Entrapped air can occur with both non-air-entrained concrete and air-entrained concrete. Entrained air and entrapped air can be distinguished by the size and shape of air bubbles.

Entrained air bubble has a typical diameter of about 50 µm [11] (between 10µm and 100µmin majority [10]). The size of entrapped air void is usually 1000µm or larger with irregular shape. It is said that entrained air can improve the workability of concrete. To achieve the similar workability, air-entrained concrete requires significantly less water and fine aggregate content comparing to non-air-entrained concrete. With the same mix-proportion, the workability of concrete will be significantly improved by increasing entrained air content.

***c) Instability of air entrainment***

Entrained airs are created by the mixing action [12] while AE agent has roles to facilitate entraining action and mainly to stabilize air bubbles in the paste [13]. Air entrainment is affected by some factors including the mixing procedure, mix-proportion, fine and coarse aggregate characteristics, physical and chemical properties of portland cement, water content and quality, dosage and properties of AE agent, other chemical admixtures, supplementary cementitious materials, and other parameters [7, 14-17].

In SCC, due to the self-compactability, obtaining an adequate air entrainment is a difficult task [18-20]. The unstable air entrainment in SCC was due to the high flowability which allowed air bubbles to move more freely thus resulted in floating of large air bubbles, fading of air bubbles diameter less than 100µm, and coalescence of air bubbles [21]. According to Fagerlund [22], mechanisms of instability of air bubbles included:

1. Large bubbles move upwards or toward the side of formwork by buoyancy force and then are lost, during transporting and compacting of concrete.
2. The collapse of bubbles by pressure arising from surface tension thus causing air bubbles dissolved in the water. This mechanism happened mostly with the smallest air bubbles explaining reason of the frequent absence of air bubbles diameter smaller than 10µm.

### 1.1.3. Air enhanced self-compacting concrete

Workability of concrete is defined as the property determining the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity, according to ASTM dictionary of engineering science and technology [23]. To enhance the workability of normal strength concrete, the self-compactability characteristic was aimed to imply to the ordinary concrete producing a new type of concrete named as air-enhanced self-compacting concrete (air-SCC). air-SCC is a normal strength concrete having self-compactability as shown in **Fig. 1.1**.

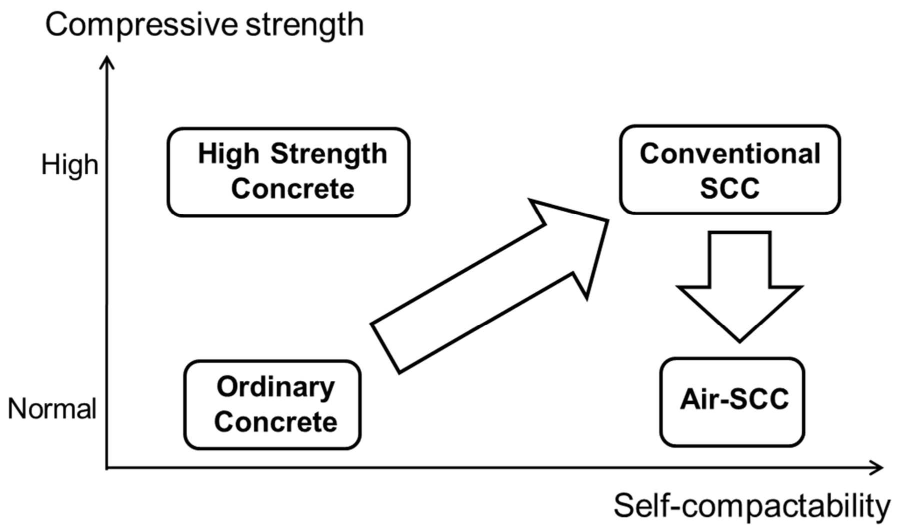



**Fig.1.1 Target for air-enhanced self-compacting concrete (air-SCC) to use in general construction**

Comparing to conventional SCC, which is the high strength concrete having self-compactability, the unit cost of air-SCC will be considerably lower. The high unit cost of conventional SCC has been the obstacle for decades in applying SCC in various kinds of construction. On the other hand, air-SCC will be able to expand the demand for SCC in the general construction.

To provide self-compactability to the ordinary concrete or to produce a normal strength SCC, adjustment on the volumetric mix-proportion was conducted on the conventional SCC. First of all, the coarse aggregate content was kept minimizing as in case of the conventional SCC. Then the volumetric mix-proportion of mortar needed to be adjusted by lowering the cement content and increasing the fine aggregate content by means of introducing higher content of air entrainment.

In this study, air-SCC was aimed produced with the water to cement ratio (W/C) of 45% by weight, the fine aggregate to mortar ratio (s/m) of 55% by volume, and the target air content of about 10%. A comparison in volumetric mix-proportion between ordinary concrete, conventional SCC, and air-SCC is shown in **Fig. 1.2**.

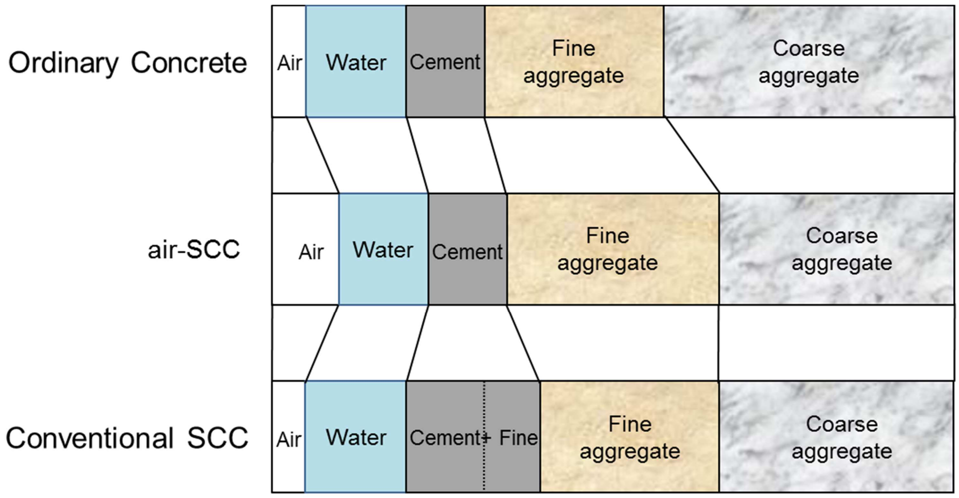



**Fig.1.2 Difference in volumetric mix-proportion of ordinary concrete, conventional SCC and air-SCC**

By comparing the volumetric mix-proportion of air-SCC to conventional SCC, the cement content was significantly reduced thus the unit cost of SCC could be reduced accordingly. By comparing the volumetric mix-proportion of air-SCC to ordinary concrete, the cement was similar which indicated that the cost of air-SCC could be similar to that of ordinary concrete.

The mix-proportion by weight and by volume portion of ordinary concrete, conventional SCC, and air-SCC are shown respectively in **Table 1.1** and **Fig. 1.3**. Unit cost of each material for concrete, currently used in this study, is shown in **Table 1.2**. The estimated unit cost of ordinary concrete, conventional SCC, and air-SCC is shown in **Table 1.3**.

**Table 1.1 Example of mix-proportion for 1m$^3$ of ordinary concrete, conventional SCC, and air-SCC**

| | Air (%) | W/C (%) | s/m (%) | (kg/1m$^3$ of concrete volume) | | | |
|---|---|---|---|---|---|---|---|
| | | | | Water* | Cement | Fine aggregate | Coarse aggregate |
| Ordinary concrete | 5 | 55 | 50 | 181 | 329 | 764 | 1018 |
| Conventional SCC | 5 | 30 | 40 | 194 | 646 | 713 | 764 |
| air-SCC | 10 | 45 | 55 | 166 | 369 | 929 | 724 |

**including SP and AE*

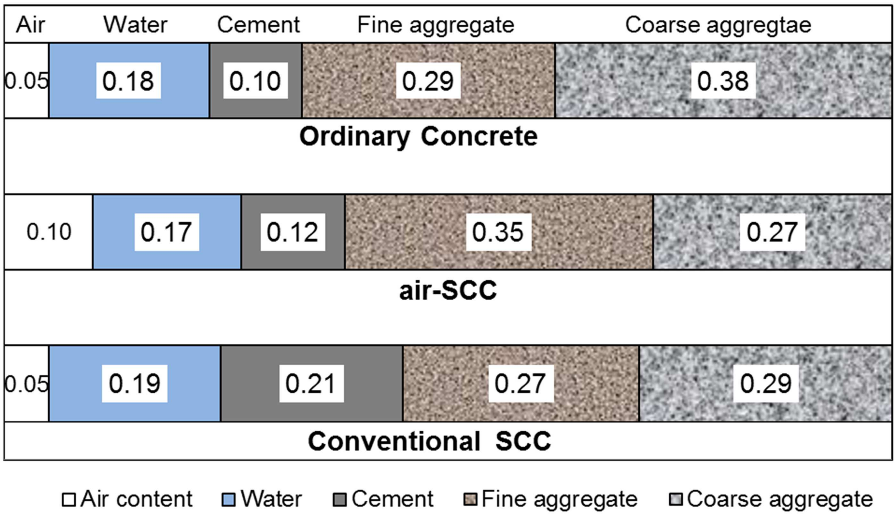



**Fig.1.3 Comparison of mix-proportion by volume between ordinary concrete, conventional SCC, and air-SCC**

**Table 1.2 Unit cost of each material used for concrete**

| Materials | Cement (C) | Fine aggregate (S) | Coarse aggregate (G) | SP | AE |
|---|---|---|---|---|---|
| Unit cost (JPY/kg) | 15 | 2-3 | 2-3 | 400 | 310 |

**Table 1.3 Comparison of unit cost of ordinary concrete, conventional SCC, and air-SCC**

| Types of concrete | Cost of each material in 1 $m^3$ concrete (JPY) | | | | | **Total cost (JPY)** |
|---|---|---|---|---|---|---|
| | C | S | G | SP | AE | |
| Ordinary concrete | 4,928 | 1,910 | 2,546 | 0 | 20 | **9,404** |
| Conventional SCC | 9,693 | 1,782 | 1,910 | 2,585 | 40 | **16,009** |
| air-SCC | 5,541 | 2,322 | 1,809 | 1,478 | 92 | **11,241** |

With a large reduction in the cement content in air-SCC, the unit cost was considerable lower than that of conventional SCC. The unit cost of air-SCC was slightly higher than that of ordinary concrete. By including the expense for concrete handling when using ordinary concrete, the final unit costs of air-SCC and ordinary concrete were comparable. With self-compactability, air-SCC will become more reliable in concrete handling for various kinds of construction projects.

## 1.2. Loss of Air Content from Fresh Mortar of air-SCC

Experiment was conducted with fresh mortar of air-SCC to measure air content just after mixing and in 2 hours after the first contact of the cement and water. A relationship between initial air content and air content in 2 hours is shown in **Fig. 1.4**.

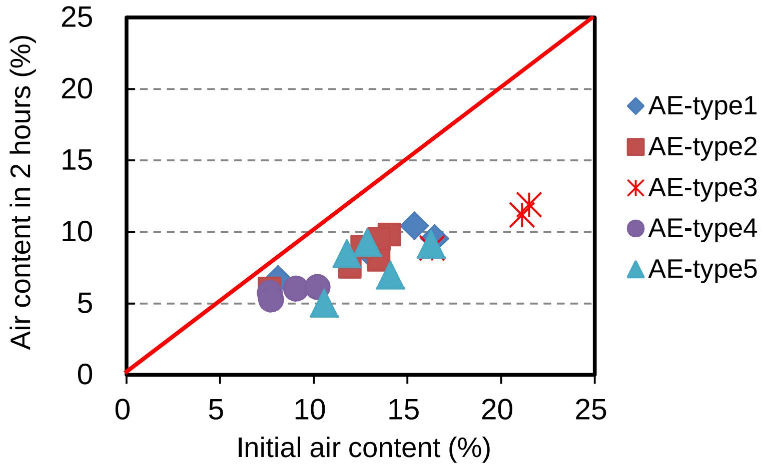



**Fig.1.4 Loss of air content in 2 hours in fresh mortar of air-SCC by using conventional mixing procedure**

According to Standard Specifications for Concrete Structure of JSCE [24], the maximum allowable time from the completion of mixing to the completion of placement of concrete should be 2 hours. It is indispensable to evaluate the quality of concrete production just before handling at construction site and that is why the air content at 2 hours was evaluated in this study. With high air content in concrete, it is more difficult task for concrete producer to estimate the variation of air from the initial stage until the completion of concrete placement. To minimize this concern, an adequate stability of air bubbles in concrete mixture needs to be ensured.

This experiment was conducted by using a conventional mixing procedure of mortar in which after the fine aggregate and the cement were mixed for 30 seconds, the mixing water, SP and AE were added and mixed for another 120 seconds. Five types of AE were used. AE-type1, AE-type2, and AE-type3 produced higher initial air content when dosage of AE was increased. AE-type4 could not produce initial air content as high as the other types of AE even with the high AE dosage. Regardless of the type of AE, higher initial air content resulted in larger loss of air content in 2 hours. Since the coarse aggregate content is limited to about 30% of concrete volume and target air content in air-SCC is about 10%, target air content needed in mortar is about 15%. From the above experimental results, the loss of air content in 2 hours around this target air content was large and not acceptable. This result also explained that achieving adequate stability of air bubbles in air-SCC could not be done simply by increasing the dosage of AE nor alternating the types of AE. Another approach needs to be established.

### 1.3. Stability of Air Bubbles

Stability of air bubbles refers to the resistance of air bubbles to deterioration. Adequate stability of air bubbles means a system of air entrainment in which air content does not increase or decrease over time. As described above, the target air content for air-SCC is about 10% so the stability of air bubbles needs to be controllable. The unpredictable loss of air content may cause a failure in the concrete design. The difficulty in assuring the stability of air bubbles in air-SCC could be partially caused by the employment of both the SP and AE at the same time in the concrete mixture. Previous studies [25-27] have reported the side effects of using SP in combination with AE including the reduction in initial air content and the unstable air entrainment. The presence of these two types of chemical admixtures in air-SCC could be the main factors affecting the stabilization of air bubbles and may hinder the workability of fresh concrete especially when they are added at the same time. The timing of the discharge of AE into the mixer is of importance so as to ensure a uniform distribution and adequate mixing for the formation of the air bubbles [11].

To ensure the stability of air in concrete is a complicated task especially with highly flowable concrete as SCC since the bubbles could move more freely causing the loss of air by either rupturing or collapsing. In SCC, the combination of SP and AE makes the mechanism of air entrainment even more complicated than that of the normal concrete. To be able to choose a better choice for improving the stability of air for SCC, basically understanding on mechanism of SP and AE is unavoidable. The mechanism of SP and AE working individually or together is described in the following section. Then mixing procedure was modified to improve the stability of air.

## 1.4. Mechanism of Superplasticizer and Air Entraining Agent

### 1.4.1. Mechanism of SP and AE acting individually in cement paste

SP is a high-range water-reducing agent working as cement dispersants through electro-steric repulsive force. Electro-steric dispersion is a combination of electrostatic and steric dispersions. According to Daimon and Roy [28], adsorption of SP on the hydrating cement hinders flocculation in three ways. The first is an increase in the zeta-potential which increased the dispersing particles (electrostatic dispersion). The second is an increase in solid-liquid affinity. When the particles are more strongly attracted to the liquid than to each other, they tend to disperse. The third is steric hindrance (steric dispersion) which is long side chain non-ionic polymer and hinder the attraction between solid particles.

SP of polycarboxylate based type (which was used in this study) is composed of polymers having a main carbon chain with carboxylate groups (electrostatic repulsion) and polyethylene oxide side chains (steric hindrance) [10]. The mechanism of SP on cement paste is shown in **Fig. 1.5**.

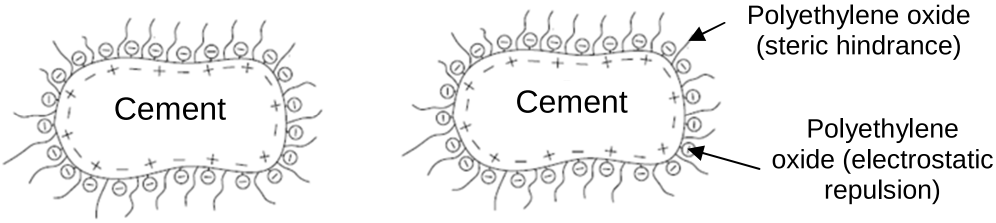



**Fig.1.5 Mechanism of SP acting in cement paste**

The polar chain is absorbed to the solid-water interface and imparts a slight negative charge causing the cement grains to repel one another. The long side chains (polyethylene oxide) physically hold the cement grains apart and keep cement dispersed when electrostatic repulsion dispersing effect wears off due to cement hydration [29].

AE is a group of surfactants which reduce the surface tension at air-water interface encouraging the formation of air bubbles. AEs help stabilizing the micro air bubbles formed during the mixing process. The basic chemical nature of surfactants is composed of two main parts: hydrophilic head and hydrophobic tail. The hydrophobic end of AE (tail) is attracted to the air and the hydrophilic end (head, usually possessed negative charges) orients itself towards the water. This action form a water-repelling film on air bubbles and the negative charged disperse air bubbles from each other. The surfactant molecules of AE adheres itself to the surface of cement particles, moving air bubbles to attach to the surface of cement particles. The mechanism of AE acting in cement paste is shown in **Fig. 1.6**.

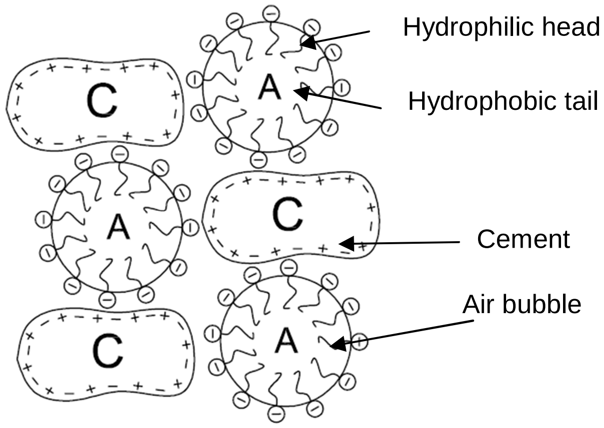



**Fig.1.6 Mechanism of AE acting in cement paste**

### 1.4.2. Mechanism of AE and SP acting together in cement paste

With the present of SP, the air content tends to be reduced. When SP and AE are poured and mixed at the same, the mechanism of these two admixtures may become more complicated. To some level, the admixtures may disturb the effectiveness of each other resulting in both poor workability of a mixture and unstable air entrainment. By considering these phenomena, the author had proposed a hypothesis in which by firstly introducing SP and mixing before introducing AE, is beneficial to allow SP to disperse the particles and lower the viscosity of the mixture. Thereafter pouring and mixing AE, a lower initial air content may be obtained but with an improvement on stability of air entrainment.

## 1.5. Preliminary Experiment: Introduction to Different Mixing Procedure on Enhancing Stability of Air

The first experiment in this chapter was to determine an effective mixing procedure which is able to enhance the stability of air bubble as much as possible. Adding to the discussion in the section **1.4**, according to Flatt and Houst [30], behavior of SP added to the mixture is divided into three components. The first part is consumed by intercalation, coprecipitation or micellization within the hydrating cement minerals which means this part of SP is no longer available for dispersing flocculated particles. This chemical reaction is significant in the first minutes after addition of SP when the precipitation rate of AFt (alumina, ferric oxide, tri-sulfate or $Al_2O_3$-$Fe_2O_3$-tri) is highest. The second part is available for adsorption to the surface of cement particles allowing the dispersing action (effective portion of SP). The third part of SP remains dissolved in the aqueous phase and may play a part in dispersing cement particles.

In the first set of experiment, the mixing procedure was modified by dividing the mixing water into two portions. The first portion was added with SP and the rest portion was added with AE. Less amount of water would reduce the increase the effective of SP molecules in dispersing the particles in the mixture. The effect of this modified mixing procedure on the stability of air bubbles will be clarified and compared to that of the simple mixing procedure.

The dividing mixing water in the mixing procedure modifying above caused complicated tasks to the ready-mixed concrete plant even though, according to the imagination above, this mixing procedure could have produced the most adequate stability of air bubble. In the second set of experiment, the mixing procedure of the effective mixing procedure was simplified by considering not dividing the mixing water. If this latest mixing procedure was effective enough to enhance the stability of air bubble, it would be recommended for using at concrete plant and would be chosen for further improvement. The target of this experiment was to seek for an effective and efficient mixing method for adequate stability of air bubbles with conveniently applicable mixing procedure. Here, the term "effective" is referred to mixing procedure which is able to produce satisfied stability of air bubbles. The term "efficient" is referred to mixing procedure which is convenient and applicable for the ready-mixed concrete plants.

### 1.5.1. Experiment for determining the effective mixing procedure for the stability of air bubbles

#### *(a) Mixing procedure for mortar*

Two different types of mixing procedure conducted in this section are simple mixing procedure (simple) and water-dividing mixing procedure (W. D.) as shown in **Fig. 1.7**.

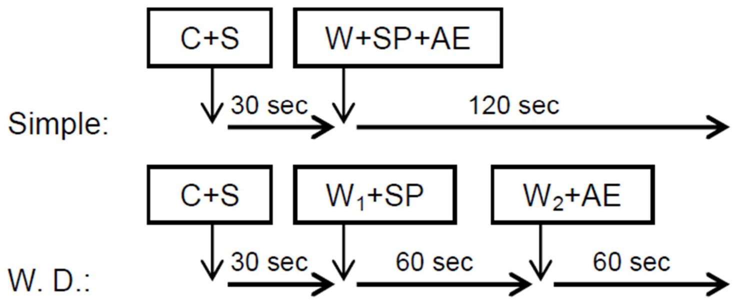



**Fig.1.7 Simple mixing procedure and water-dividing mixing procedure for mortar**

In the simple procedure, the fine aggregate and the cement were firstly mixed for 30 seconds. Then, the mixing water, SP, and AE were poured and mixed for another 120 seconds. In the W.D. mixing procedure, the mixing water was divided into two portions in which the first portion was corporate with SP and the rest part was with AE. The dividing water was based on the explanation above. In his mixing procedure, after the fine aggregate and the cement were mixed for 30 seconds, the first portion of mixing water and SP were poured and mixed for 60 seconds. And finally, the rest portion of mixing water and AE were poured and mixed for another 60 seconds. The first portion of mixing water was primarily fixed at 20% of cement weight to keep the same characteristic of the mixture before adding AE even when the dosage of AE was varied. 20% of cement weight was around half of the total mixing water since the W/C was 45% by weight (W means water including SP and AE).

***(b) Materials and mix-proportion***

A mixer with a rotation speed of 140±5 rpm was used for mixing mortar for the tests in this whole study. The materials used in this chapter are shown in **Table 1.4**.

**Table 1.4 Materials used for mortar experiment**

| | | |
|---|---|---|
| Cement (C) | | Ordinary Portland cement (3.15g/cm$^3$) |
| Fine aggregate (S) | | Crushed limestone sand (2.68g/cm$^3$, F. M. 2.73) |
| Superplasticizer (SP) | $SP_1$ | Polycarboxylic based with viscosity agent |
| | $SP_3$ | Polycarboxylic based with viscosity agent and retarding type |
| Air entraining agent (AE) | $AE_1$ | Alkyl ether-based anionic surfactants<br>*(AE: water concentration is 1:99)* |
| | $AE_2$ | Vinsol resin*(AE: water concentration is 1:24)* |

In this chapter, the W/C and s/m were fixed at 45% by weight and 55% by volume respectively. The mix-proportion for the first set of experiments in this chapter is shown in **Table 1.5**.

**Table 1.5 Mix-proportion of mortar for two different types of mixing procedure**

| W/C (% by weight) | s/m (% by volume) | $SP_3$/C (%) | AE/C (%) | Mixing procedure |
|---|---|---|---|---|
| 45 | 55 | 1.4 | 0.005 to 0.030 ($AE_1$) | Simple |
| 45 | 55 | 1.4 | 0.016 to 0.120 ($AE_2$) | Simple |
| 45 | 55 | 1.4 | 0.005 to 0.050 ($AE_1$) | W. D. |
| 45 | 55 | 1.4 | 0.016 to 0.200 ($AE_2$) | W. D. |

The dosage of SP was 1.4% of cement weight for all mixes. In the case of simple mixing procedure, the dosage of AE was varied from 0.005% to 0.030% of cement weight and from 0.016% to 0.120% of cement weight in case of $AE_1$and $AE_2$ respectively. In the case of W. D. mixing procedure, the dosage of AE was varied from 0.005% to 0.060% of cement weight and from 0.016% to 0.240% of cement weight in case of $AE_1$ and $AE_2$ respectively.

### 1.5.2. Experiment for determining the effective and efficient mixing procedure for the stability of air bubbles

#### *(a) Mixing procedure for mortar*

The first mixing procedure was the simple mixing procedure (named here as mixing procedure A) and the second mixing procedure was the W. D. mixing procedure (named here as mixing procedure B). The third mixing procedure was mixing procedure C, as shown in **Fig. 1.8**.

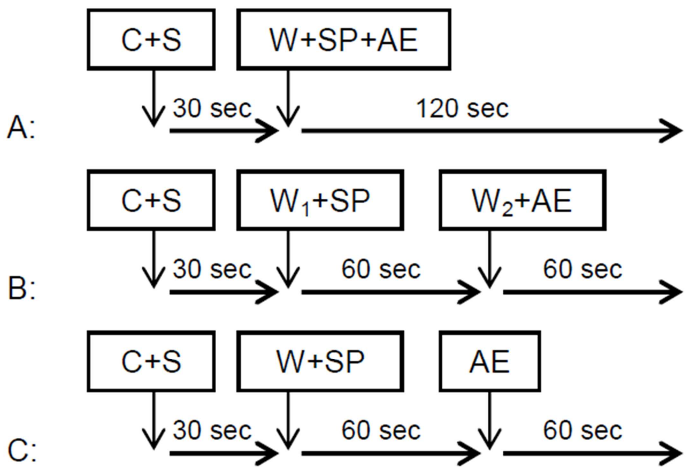



**Fig.1.8 Different types of mixing procedure for mortar**

In this latest mixing procedure, SP and AE were poured separately from each other but the mixing water was not divided. First of all, the cement and the fine aggregate were mixed for 30 seconds. Then all the mixing water and SP were poured and mixed for 60 seconds. Finally, AE was poured and mixed for the last 60 seconds.

#### *(b) Materials and mix-proportion*

The materials used in this section can be found in **Table 1.4**. The mix-proportion for the second set of experiments in this chapter is shown in **Table 1.6**.

**Table 1.6 Mix-proportion of mortar for three different types of mixing procedure**

| W/C (% by weight) | s/m (% by volume) | $SP_1/C$ (%) | $AE_1/C$ (%) | Mixing procedure |
|---|---|---|---|---|
| 45 | 55 | 1.2 | 0.005 to 0.100 | A |
| 45 | 55 | 1.2 | 0.005 to 0.100 | B |
| 45 | 55 | 1.2 | 0.005 to 0.100 | C |

The dosage of SP was 1.2% of cement weight for all mixes. The dosage of AE was varied from 0.005% to 0.100% of cement weight for each mixing procedure.

### 1.5.3. Effective mixing procedure: water-dividing mixing

The relationship between dosage of AE and initial air content produced by the simple mixing procedure and the W. D. mixing procedure is shown in **Fig. 1.9**.

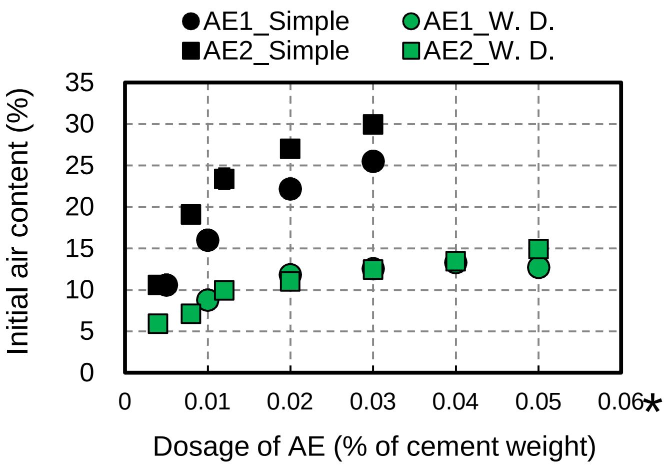



**Fig.1.9 Effect of water-dividing mixing procedure (W. D.) on initial air content**

*(*dosage of AE ×4 in case of $AE_2$)*

The W.D. mixing procedure produced considerably lower initial air content than that of the simple mixing procedure regardless the types of AE. This result was in accordance with the hypothesis mentioned above. In case of the W. D. mixing procedure, due to lower in viscosity, less air bubbles were able to be generated. As a result, initial air content obtained with the W. D. mixing procedure was lower than that obtained with the simple mixing procedure. It can be seen that, to reach same target air content, the dosage of AE required in case of W. D. mixing procedure was significantly higher than that required by the simple mixing procedure. The requirement of AE dosage could be an important factor for W. D mixing procedure in entraining finer air size distribution.

The relationship between dosage of AE and the variation in air content in 2 hours is shown in **Fig. 1.10**.

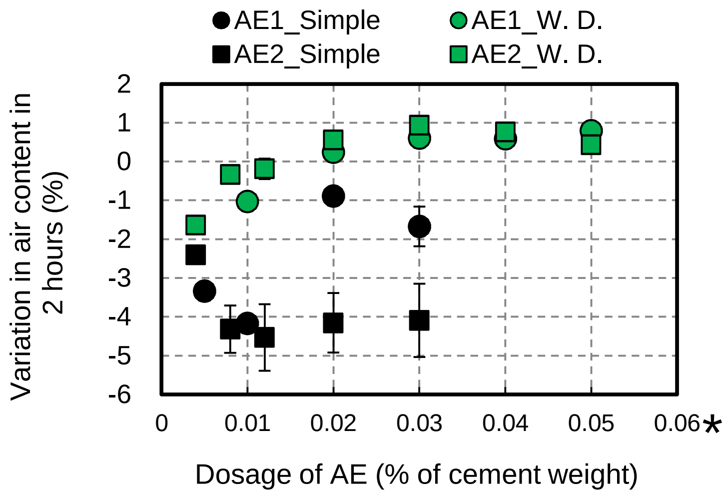



**Fig.1.10 Effect of water-dividing mixing procedure (W. D.) on stability of air bubbles**

*(*dosage of AE ×4 in case of AE$_2$)*

Negative value means the air content was reduced. The loss of air content was large in case of the simple mixing procedure and was not able to be improved with the increase dosage of AE. On the other hand, with the W. D. mixing procedure, the stability of air bubbles was considerably satisfied. The improvement of the stability of air bubbles can also be observed with the increase in dosage of AE. This result proved that modifying the mixing procedure effectively influenced the stability of air bubbles and that by adding SP before AE, the significant improvement of the stability of air bubbles could be achieved.

Funnel speed of fresh mortar ($R_m$) is defined from the flow-through time of the V-funnel test as shown in **Fig. 1.11** and in **Eq. 1**.

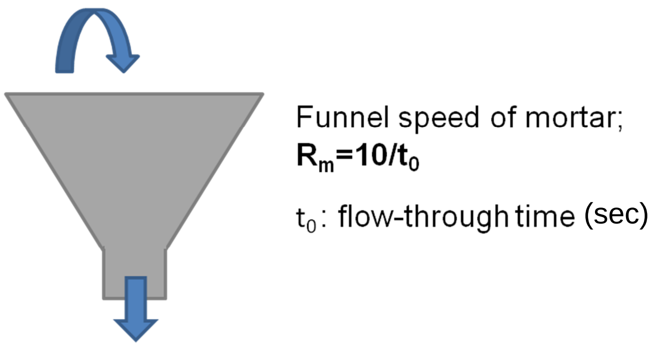



**Fig.1.11 Test for funnel speed of mortar**

$$R_m = \frac{10}{t_0};$$ **Eq. 1**

where $t_0$ is flow-through time of mortar (second)

This index was considered as it is partially able to indicate the viscosity of the mortar mixture. And interestingly, since the modifying on mixing procedure was done by considering the dispersing action of SP, the funnel speed of mortar produced by different mixing procedure could be significantly different.

The relationship between dosage of AE and funnel speed of mortars produced with simple mixing procedure and W. D. mixing procedure is shown in **Fig. 1.12**.

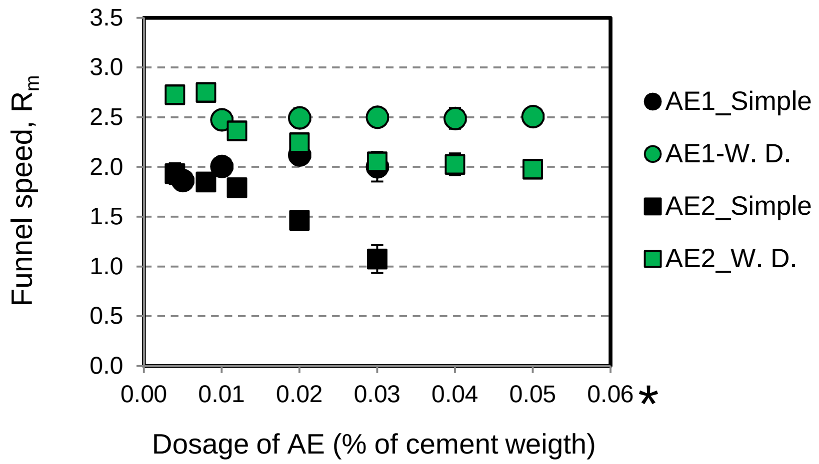



**Fig.1.12 Effect of dosage and type of AE on funnel speed of mortar ($R_m$) produced with simple procedure and W. D. mixing procedure**

*(*dosage of AE ×4 in case of $AE_2$)*

With either type of AE, funnel speed of mortars produced with W. D. mixing procedure was higher than that of simple mixing procedure. Different type of AE resulted in different tendency of funnel speed when AE dosage was increased. With $AE_1$, with both mixing procedure, the funnel speed was not significantly varied when AE dosage was increased. With $AE_2$, with both mixing procedures, the funnel speed was decreased when AE dosage was increased. This result was influenced by the composition of the AE which determined different nature to each AE.

### 1.5.4. Effective and efficient mixing procedure

The relationship between dosage of AE and initial air content produced by mixing procedure A, B, or C is shown in **Fig. 1.13**.

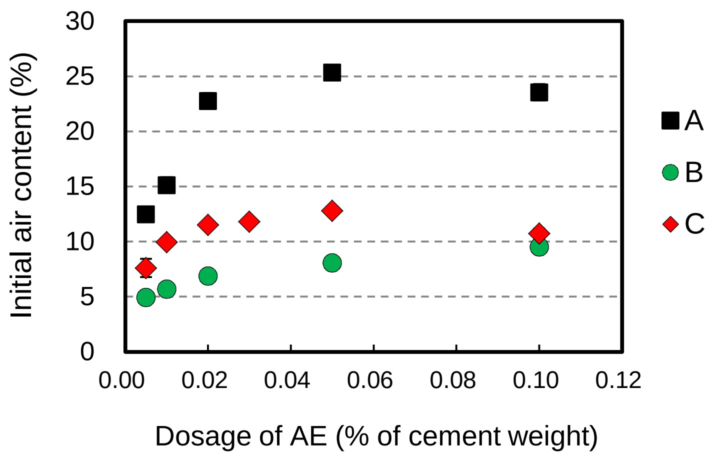



**Fig.1.13 Effect of different mixing procedures on initial air content**

At any dosage of AE, mixing procedure A produced the highest initial air content among these mixing procedures. Based on the hypothesis above, the mechanism of AE and SP acting in case of method B or C was in similar way since SP was added before AE. Initial air content produced by mixing procedure C was slightly higher than that produced with mixing procedure B. In case of mixing procedure C, SP deserved for dispersing action was lower than that in case of mixing procedure B. The difference in capacity in entraining air volume was due to the dispersion between cement particles and water resulting in difference in viscosity in each mixture.

The relationship between dosage of AE and the variation in air content in 2 hours produced by mixing procedure A, B, and C is shown in **Fig. 1.14**.

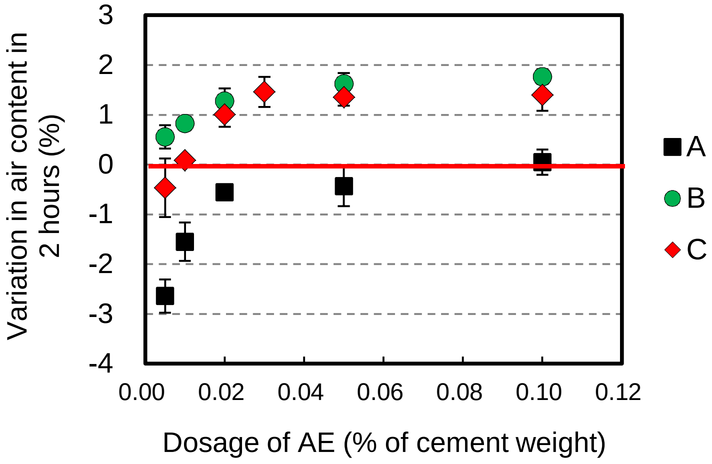



**Fig.1.14 Effect of different types of mixing procedure on stability of air bubbles**

Mixing procedure B or C produced better stability of air bubbles than mixing procedure A. However, the increase in air content in 2 hours was occurred in case of mixing procedure B or C. When the mixture was disturbed by re-mixing before the test could cause extra air bubbles to be created if the dosage of AE was sufficient for stabilizing those newly formed air bubbles.

The relationship between dosage of AE and funnel speed of mortars produced with mixing procedure A, B or C is shown in **Fig. 1.15**.

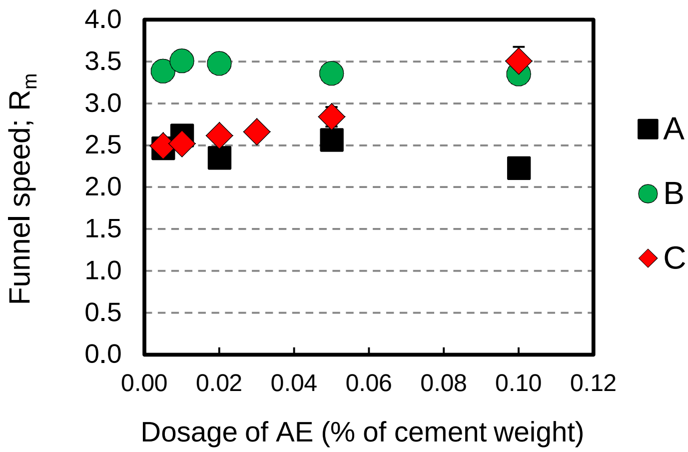



**Fig.1.15 Effect of AE dosage on funnel speed of mortar ($R_m$) produced with different types of mixing procedure**

Funnel speed of mortars produced with mixing procedure B was significantly higher than that of mixing procedure A. While the funnel speed of mortar produced with mixing procedure A or B was not varied with the increase dosage of AE, the funnel speed of mortar produced with mixing procedure C accordingly increased with the increase dosage of AE. It was interesting to observe that the funnel speed of mortar produced with mixing procedure C was similar to that of mixing procedure A at low dosage of AE and was similar to that of mixing procedure B at high dosage of AE. At low dosage of AE, the total mixing water firstly poured with SP in case of mixing procedure C was close to that of mixing procedure A (normally a part of mixing water was used for concentrating AE as recommendation from the producer). The mixing water firstly poured with SP in case of mixing procedure C decreased accordingly with the increase dosage of AE; therefore at high dosage of AE, mixing procedure C acted similarly to mixing procedure B. The amount of water firstly poured with SP was a significant parameter determined the dispersing action of the mixture as well as the funnel speed.

## 1.6. Observation on Air Size Distribution in Mortar at Hardened Stage Measured with Linear Traverse Method

### 1.6.1. Linear traverse method

***a) Significance of test method***

Air size distribution of hardened mortar samples in this study was tested following the ASTM C 457 (standard test method for microscopical determination of parameters of air-void system in hardened concrete) procedure A [31]. Linear traverse method consists of the determination of air volume of the concrete by summing the length traversed across air bubbles along a series of regularly spaced lines in the plane intersecting the sample. The significance of this method is to enable developing data to estimate the likelihood of damage due to the cycle of freezing and thawing or to explain why it has occurred. In this study, data generated from this test method will enable explaining the effect of different mixing procedure and different mix-proportion on the stability of air bubbles.

***b) Terminology and calculation***

The data gathered in this test method are the total length traversed ($T_t$), the length traversed through air bubbles ($T_a$), and the number of air voids intersected by the traverse line (N). The parameters calculated from these data include the total air content, the specific surface, and the average chord length following the **Eq. 2**, **Eq. 3**, and **Eq. 4** respectively. The total air content is defined as the proportion of total volume of the air voids to that of the mixture; expressed in % by volume. The specific surface is the surface area of the air bubbles in a unit volume of air bubble; expressed in $mm^2/mm^3$. The specific surface of air can be used to evaluate the fineness of air bubbles. Total surface area of air is proportional to the specific area and the total air content; expressed in $mm^2/mm^3 of\ the\ mixture$.

Total air content; %

$$A = \frac{T_a.100}{T_t} \qquad \textbf{Eq. 2}$$

Specific surface of air; $mm^2/mm^3$

$$\alpha = \frac{4}{\bar{l}} \qquad \textbf{Eq. 3}$$

which $\bar{l}$ is called the average chord length; $mm$

$$\bar{l} = \frac{T_a}{N} \qquad \textbf{Eq. 4}$$

***c) Samples preparation***

Mortar samples were casted in the cylinder mold with diameter of 100 mm and height of 200mm. The mold was removed 24 hours after cement and water were first at contact. Then all the samples were cured in the water before cutting process. Three pieces were extracted from each mortar sample with the thickness of 50mm as shown in **Fig. 1.16**. All the pieces were polished and then gently cleaned before air-voids distribution test by linear traverse method. The investigated area was 60×60 mm and the total traversed length was 2615.9 mm in all measurements. The photos of air-voids distribution of concrete samples tested by linear traverse method are shown in **Fig. 1.17**.

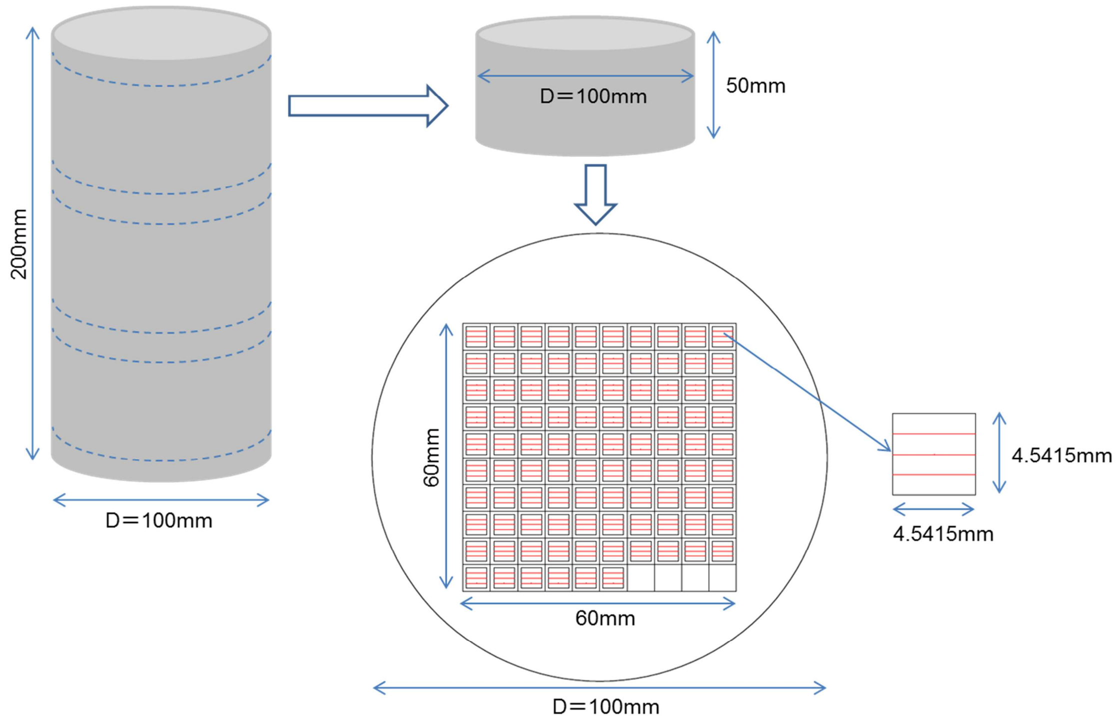



**Fig.1.16 Mortar sample preparation for linear traverse method test**

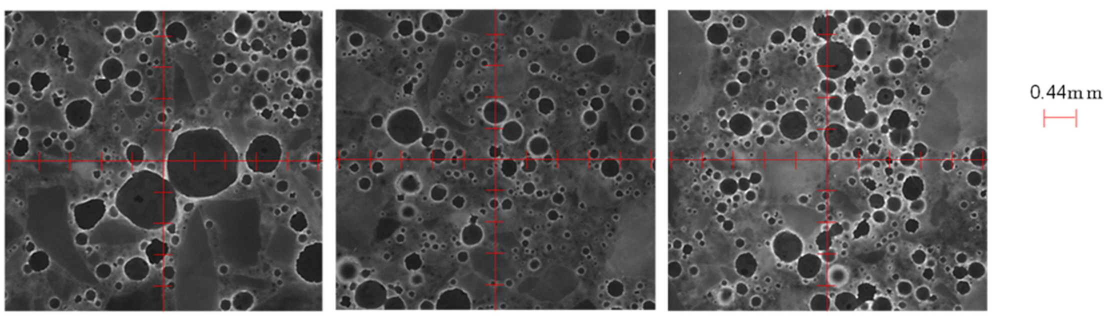



**Fig.1.17 Photos of air size distribution of concrete samples produced by different mixing procedure and tested by linear traverse method**

### 1.6.2. Observation on correlation between stability of air and air volume at different border of bubbles size

***a) Experimental plan***

To observe the influence of air bubbles at different size on the stability of air, air size distribution in mortar were measured firstly by linear traverse method at hardened stage. The size of air bubbles could be a significant factor to the stability of air because coarse air bubbles are more likely to be escaped faster than the smaller ones. The critical size of air bubble is defined as the size of air which easily to be escaped and harmfully affects the stability of air.

The target of this section is to determine a critical size of air bubbles to the stability of air. The total of 21 mortar samples was selected for air bubbles distribution test using linear traverse method. These samples were made with different combination of mixing procedure, mixing time, dosage of SP or AE, and type of SP or AE. The mix-proportion and the properties at initial stage of these samples are shown in **Table 1.7**. The properties of materials used in this study are shown in **Table 1.8**.

**Table 1.7 Mix-proportion and properties of mortar samples tested with LTM**

| N° | Mixing procedure | SP/C (%) | | AE/C (%) | | Flow (mm) | Funnel speed | Air content (%) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Gravitic | | LTM | |
| | | | | | | | | $A_{initial}$ | $A_{2h}$ | $A_{hardened}$ | $A_{>700\mu m}$ |
| 1 | $A_2$ | $SP_1$ | 1.4 | $AE_2$ | 0.040 | 259 | 2.35 | 17.5 | 14.4 | 12.8 | 3.81 |
| 2 | $A_2$ | $SP_1$ | 1.4 | $AE_2$ | 0.200 | 181 | 1.56 | 29.4 | 24.6 | 22.0 | 6.19 |
| 3 | $B_{1,1}$ | $SP_1$ | 1.4 | $AE_2$ | 0.040 | 270 | 3.27 | 7.0 | 8.1 | 7.1 | 0.99 |
| 4 | $B_{1,1}$ | $SP_1$ | 1.4 | $AE_2$ | 0.200 | 259 | 3.18 | 8.9 | 10.6 | 8.8 | 0.45 |
| 5 | $C_{1,1}$ | $SP_1$ | 1.4 | $AE_2$ | 0.040 | 268 | 2.26 | 9.8 | 10.4 | 10.6 | 2.71 |
| 6 | $C_{1,1}$ | $SP_1$ | 1.4 | $AE_2$ | 0.200 | 265 | 2.46 | 11.3 | 13.1 | 12.0 | 1.08 |
| 7 | $C_{1,1}$ | $SP_1$ | 1.4 | $AE_2$ | 0.080 | 235 | 2.35 | 13.1 | 14.0 | 14.3 | 3.00 |
| 8 | $C_{1,2}$ | $SP_1$ | 1.4 | $AE_2$ | 0.080 | 239 | 2.39 | 17.2 | 17.4 | 17.4 | 2.54 |
| 9 | $C_{1,3}$ | $SP_1$ | 1.4 | $AE_2$ | 0.080 | 250 | 2.27 | 21.0 | 20.4 | 17.3 | 3.13 |
| 10 | $C_{2,2}$ | $SP_1$ | 1.4 | $AE_2$ | 0.080 | 261 | 2.66 | 15.3 | 16.0 | 15.6 | 2.16 |
| 11 | $C_{0,2}$ | $SP_2$ | 0.9 | $AE_2$ | 0.080 | 156 | 1.32 | 27.9 | 21.4 | 18.8 | 5.32 |
| 12 | $C_{0.5,2}$ | $SP_2$ | 0.9 | $AE_2$ | 0.080 | 233 | 2.02 | 19.0 | 18.3 | 15.3 | 3.10 |
| 13 | $C_{1,2}$ | $SP_2$ | 0.9 | $AE_2$ | 0.080 | 255 | 2.50 | 16.2 | 16.0 | 14.3 | 2.17 |
| 14 | $C_{2,2}$ | $SP_2$ | 0.9 | $AE_2$ | 0.080 | 261 | 2.62 | 17.3 | 17.3 | 16.2 | 2.48 |
| 15 | $C_{2,2}$ | $SP_2$ | 0.5 | $AE_1$ | 0.015 | 184 | 2.31 | 18.3 | 17.9 | 17.0 | 3.92 |
| 16 | $C_{2,2}$ | $SP_2$ | 0.7 | $AE_1$ | 0.015 | 245 | 2.82 | 17.9 | 18.1 | 17.3 | 0.81 |
| 17 | $C_{2,2}$ | $SP_2$ | 0.8 | $AE_1$ | 0.015 | 261 | 2.98 | 17.0 | 17.2 | 15.9 | 0.83 |
| 18 | $C_{1,3}$ | $SP_1$ | 1.3 | $AE_1$ | 0.015 | 264 | 3.09 | 15.2 | 15.1 | 13.9 | 1.32 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 19 | $C_{2,2}$ | $SP_1$ | 1.4 | $AE_2$ | 0.060 | 265 | 2.63 | 15.1 | 15.5 | 15.5 | 2.72 |
| 20 | $C_{2,1.75}$ | $SP_2$ | 0.8 | $AE_1$ | 0.013 | 266 | 3.10 | 15.0 | 14.9 | 13.9 | 1.49 |
| 21 | $C_{2,2}$ | $SP_2$ | 1.0 | $AE_2$ | 0.080 | 268 | 2.83 | 15.3 | 15.0 | 13.5 | 2.48 |

*In all cases, W/C=45% by weight and s/m=55% by volume*

**Table 1.8 Materials used for mortar experiments for air size distribution**

| | |
|---|---|
| Cement (C) | Ordinary Portland cement (3.15g/cm$^3$) |
| Fine aggregate (S) | Crushed limestone sand (2.68g/cm$^3$, F. M. 2.96) |
| Superplasticizer (SP) | $SP_1$: Polycarboxylic based with viscosity agent |
| | $SP_2$: Polycarboxylic based |
| Air-entraining agent (AE) | $AE_1$: Alkyl ether-based anionic surfactants *(AE: water concentration is 1:99)* |
| | $AE_2$: Vinsol resin*(AE: water concentration is 1:24)* |

The mixing procedures using in this section are shown in **Fig. 1.18**.

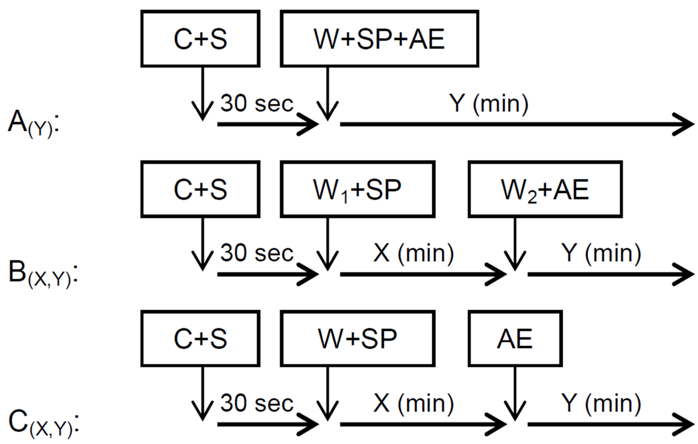


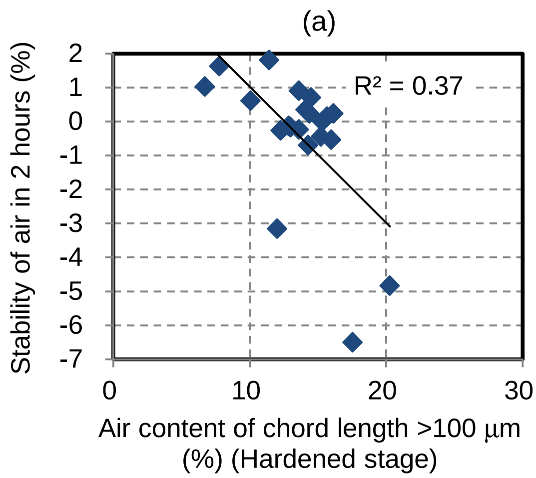

**Fig.1.18 Mixing procedures for mortar conducted in this section**

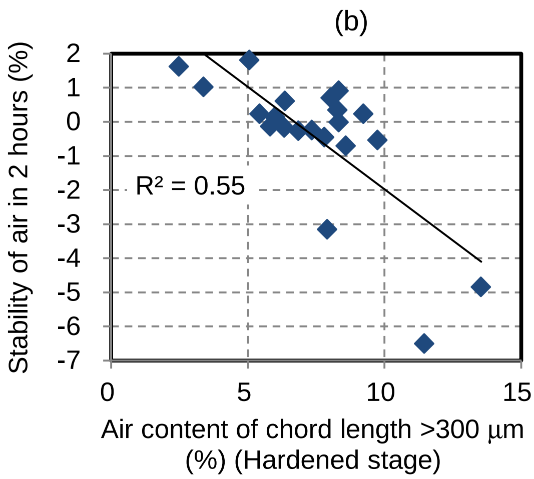
In mixing procedure $A_{(Y)}$, after the cement and the fine aggregate were mixed for 0.5 minute, the mixing water, SP and AE were poured and mixed for Y minutes. In this section, Y was 2 minutes in case of mixing procedure A. In mixing procedure $B_{(X,Y)}$, the mixing water was divided into two portions. After the cement and the fine aggregate were mixed for 0.5 minute, the first portion of water was poured with SP and mixed for X minutes. Then the rest portion of water was poured with AE and mixed for Y minutes. For mixing procedure $B_{(X,Y)}$ in this section, X and Y were chosen to be 1 minute and the first portion of mixing water was fixed at 20% of cement weight. In mixing procedure $C_{(X,Y)}$, after the cement and the fine aggregate were mixed for 0.5 minute, the mixing water and SP were poured and mixed for X minutes. Finally, the AE was poured and mixed for Y minutes. In this case with mixing procedure $C_{(X,Y)}$, X was varied at 0, 0.5 minute, 1 minute and 2 minutes. Y was varied at 1 minute, 2 minutes, 3 minutes and 1.75 minutes. The different procedure of mixing could significantly change the properties of air entrainment system. Thus, by varying the mix-proportion with different mixing procedures, various group of air entrainment could be collected to evaluate the critical of air bubbles which is harmful to the stability of air bubbles.

***b) Results***

At fresh stage, air content of each mortar sample was measured by gravitic method, just after mixing and in 2 hours after the first contact of water and cement. At hardened stage, air content was determined by linear traverse method. The variation in air content from the initial stage until hardening for all 21 mortar samples can be found in **APPENDIX A**. The size distribution of air bubbles is indicated with its chord length which, according to ASTM C 457, is assumed to equal 2/3 of its diameters.

The relationship between the stability of air and the air content at hardened stage for each chord length range (>100 µm, >300 µm, >500 µm, >600 µm, >700 µm, >800 µm, >1000 µm, or >1500 µm) are shown respectively in **Fig.1.19**. Among these eight figures, the highest value of correlation ($R^2$) was obtained with chord length of air bubble of larger than 700 µm, which is 0.68.



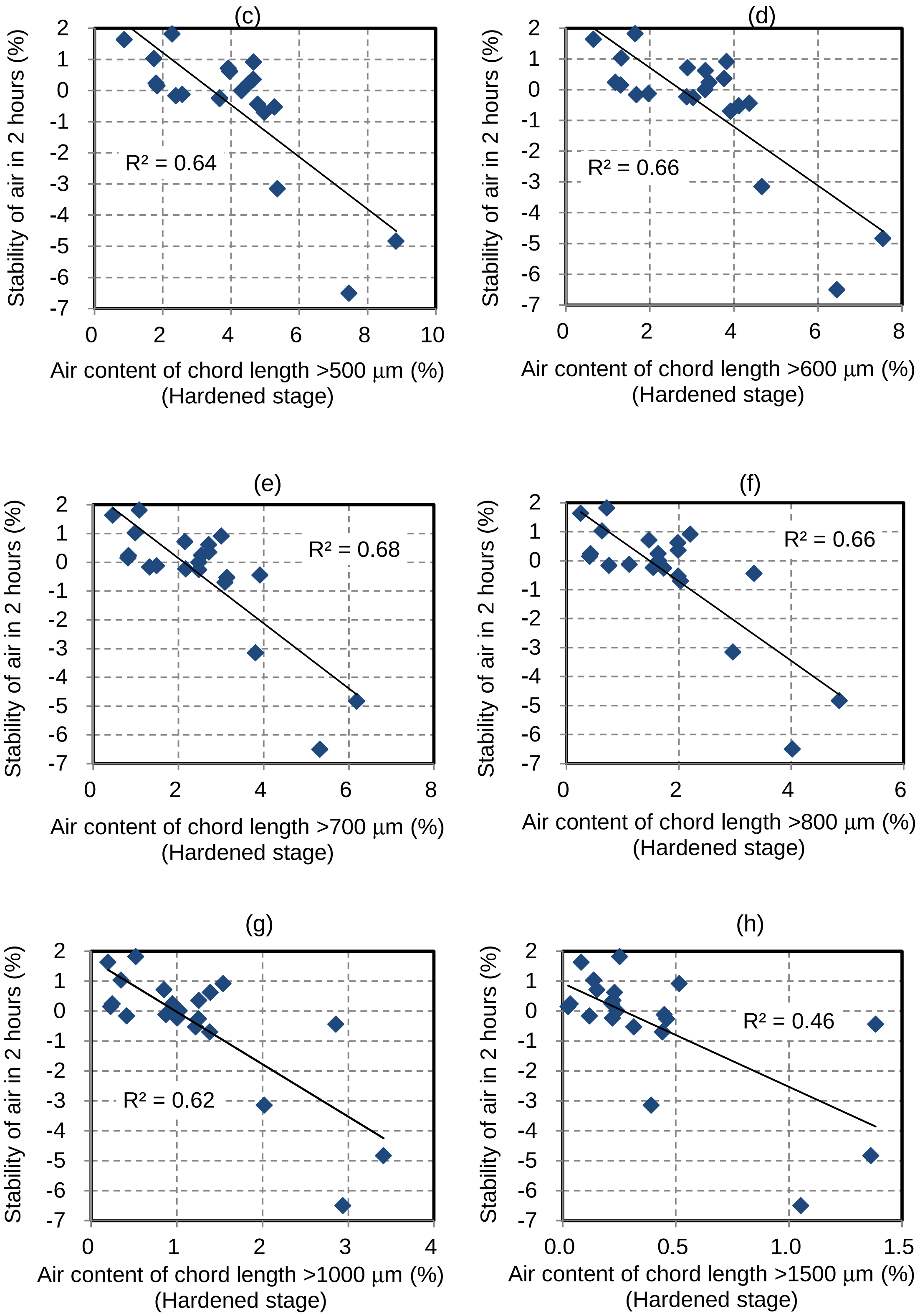



**Fig.1.19 Relationship between the stability of air in 2 hours and the air content at hardened stage of chord length (a): >100 μm, (b): >300 μm, (c): >500 μm, (d): >600 μm, (e): >700 μm, (f): >800 μm, (g): >1000 μm, or (h): >1500 μm**

The correlation between the stability of air and the air content at hardened stage for each chord length range of air bubbles is shown in **Fig. 1.20**.

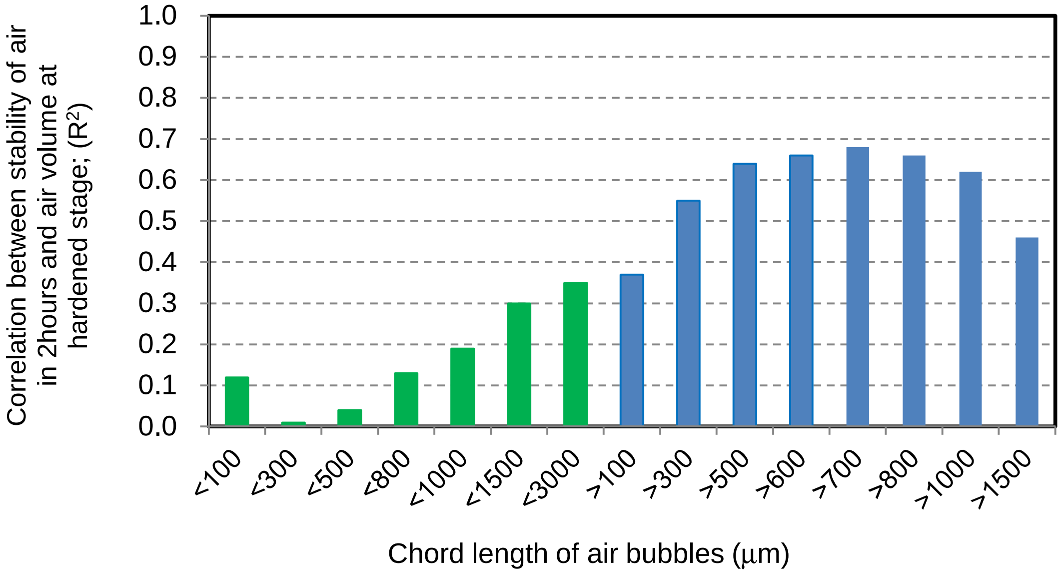



**Fig.1.20 Correlation between the stability of air in 2 hours and air volume at hardened stage of different border of bubbles size**

The graph explained that the stability of air depended on the air volume of coarser size bubbles rather than the smaller ones. The highest value of correlation ($R^2$) was obtained at the chord length of larger than 700 µm. This result indicated that the coarse size air bubbles were not favorable for the stability of the air-voids system.

However, these correlations were obtained by using the air content measured at hardened stage which was not totally represented initial air-void system just after mixing. The actual critical size could be larger than 700 µm since it was more likely that the coarser air bubbles could escaped faster than the smaller ones during fresh stage and also hardening. This can explain that some portion of coarser air bubbles was already lost before air distribution test at hardened stage. Anyway, the air distribution and the determination on the critical size of air bubbles on the stability of air will be done in the next chapter at fresh stage by air-void analyzer which is expected to bring a more precise result on the critical value.

***c) Observation 1: Effective mixing procedure minimized the coarse size air bubbles***

At the same dosage of AE of 0.20% of cement weight, the relationship between initial air content and the stability of air content in 2 hours of the mortar samples produced with different mixing procedure A, B or C is shown in **Fig. 1.21**.

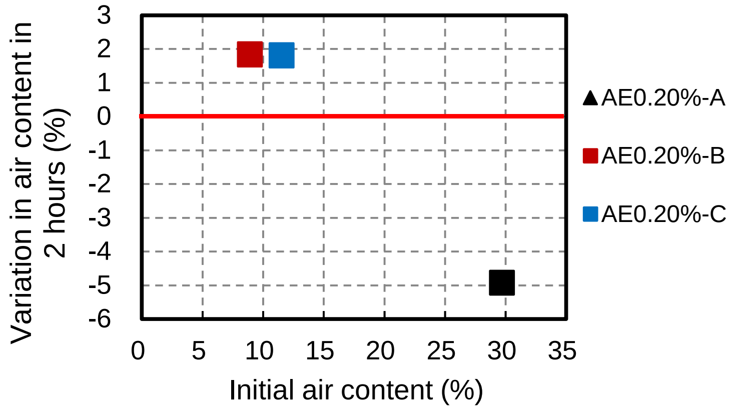



**Fig.1.21 Effect of mixing procedure on stability of air bubbles**

Initial air content produced with mixing procedure A was much higher than that of mixing procedure B or C. However, the stability of air bubbles produced with mixing procedure B or C was much better than that of mixing procedure A. With mixing procedure B or C, there could be more room for improvement the volume of air accompanying with the finer system of air entrainment.

Air size distribution in mortar samples produced with mixing procedure A, B, or C with the same mix-proportion at AE dosage of 0.20% of cement weight is shown in **Fig. 1.22**.

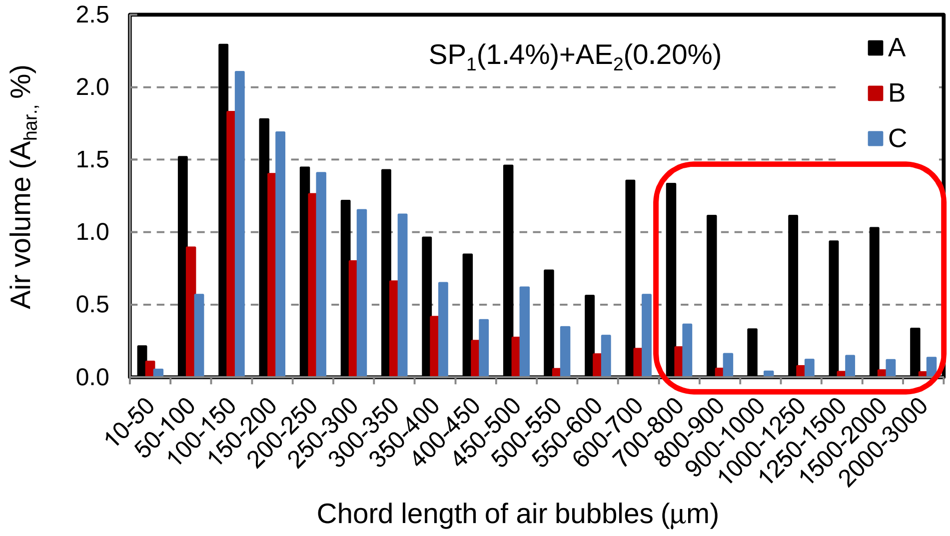



**Fig.1.22 Air size distribution in mortars produced with mixing procedure A, B, or C with AE dosage of 0.20% of cement weight**

When the dosage of AE increased, the air bubbles distribution of mixing procedure B or C was significantly improved. The content of coarse size air bubbles was almost eliminated with mixing procedure B or C. The increase of AE dosage lowered the surface tension then encouraging more air bubbles to be created. With a high flowability of the mixture produced by mixing procedure B or C (by dispersing action), the coarse size air bubbles were difficult to be formed or some of them were already escaped before mixing was finished. This reason also explained why even though the dosage of AE was increased to be very high, the air content was not accordingly increased when mixing procedure B or C was conducted. On the other hand, with the high increase in air content with mixing procedure A when AE dosage was increased, there was no improvement on the air bubbles distribution observed since the content of coarse size air bubbles was also increased in this case.

The share of air volume at five ranges of chord length of air bubbles are shown in **Fig. 1.23**.

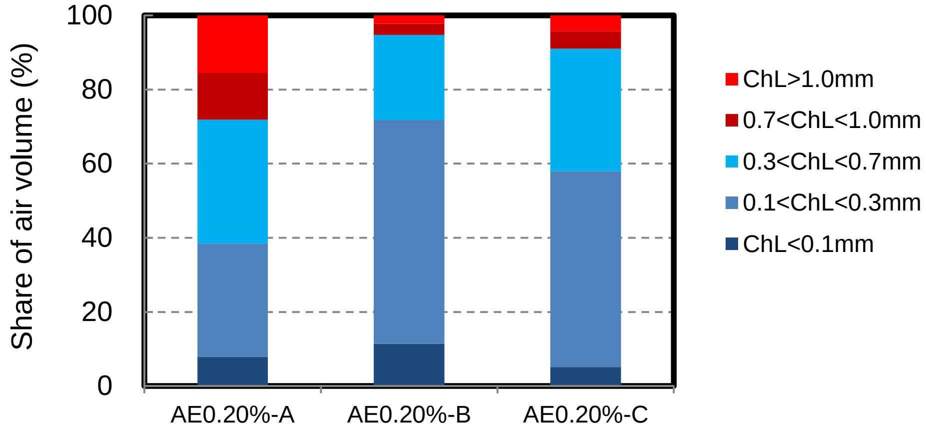



**Fig.1.23 Share of air volume at each range of chord length (ChL) of air bubbles in mortar produced with mixing procedure A, B or C**

The share of air volume of bubble with chord length of smaller than 700 μm of mixing procedure B or C was significantly larger than that of mixing procedure A. This result showed that the air bubbles distribution of mortar produced with mixing procedure B or C was finer than that produced by mixing procedure A.

A finer air-void system found with mixing procedure B than with mixing procedure C could be related to the flowability of the mortar samples produced with these two types of mixing procedure. The funnel speed of mortar ($R_m$) produced with mixing procedure B was faster than that produced with mixing procedure C. The relationship between the funnel speed and the specific surface of air is shown in **Fig. 1.24**.

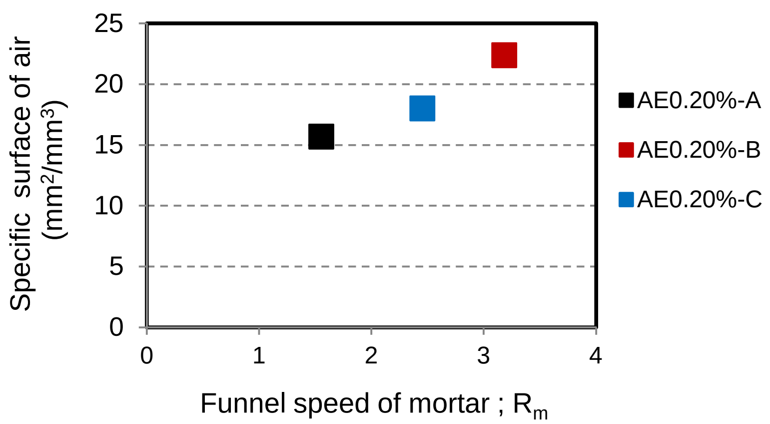



**Fig.1.24 Relationship between funnel speed and the specific surface of air of mortars produced with different types of mixing procedure**

With the same dosage of AE, higher funnel speed was accompanied with finer air-void system. This was the result of the dispersing action of SP before AE was added in case of mixing procedure B or C that lead to the finer air-void system than in case of mixing procedure A. With mixing procedure B or C, the air volume of coarse bubble could be interestingly minimized along with the lower viscosity value as shown in **Fig. 1.25**.

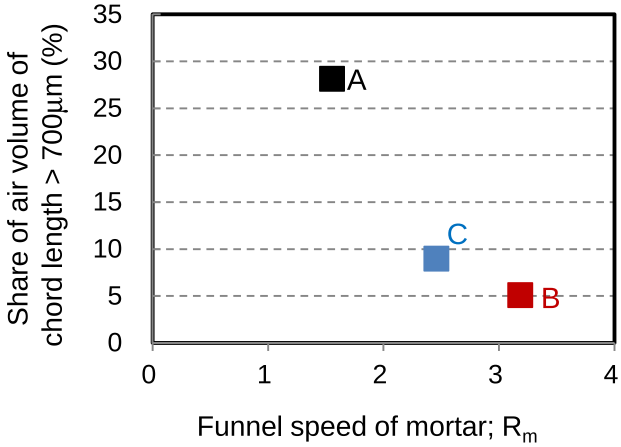



**Fig.1.25 Relationship between funnel speed and the share of coarse air volume of mortars produced with different types of mixing procedure**

***d) Observation 2: Mixing time with AE increased the total air volume while mixing time with SP reduced air volume of coarse bubbles***

Three pairs of mortar samples with the same mix-proportion produced with mixing procedure $C_{1,Y}$ in which the mixing time with SP was 1 minute and the mixing time with AE was varied by parameter "Y" at 1 minute, 2 minutes, or 3 minutes respectively. The effect of mixing time with AE on initial air content and stability of air bubbles in 2 hours is shown in **Fig. 1.26**.

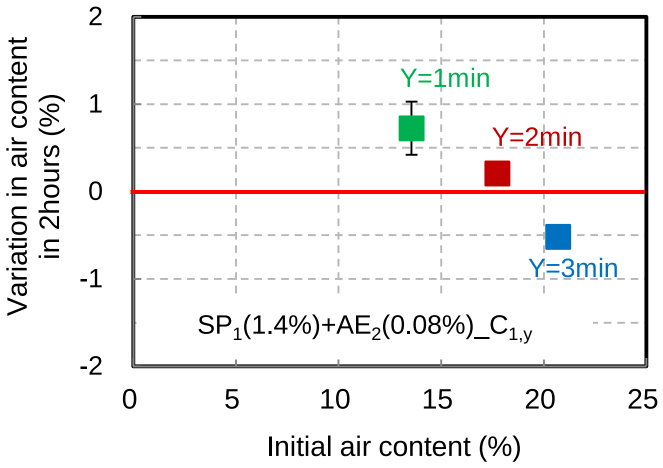



**Fig.1.26 Effect of mixing time with AE on initial air content and stability of air bubbles in 2 hours**

With longer mixing time with AE, initial air content increased whereas the stability of air slightly was hindered. This result can be explained that longer mixing time with AE provided more chance for air bubbles to be created resulting in higher content of air. However, with the same dosage of AE, longer mixing time also allow more air bubbles to be coalesced and formed a new larger size air bubbles. At the same time as small size air bubbles increased with longer mixing time, the content of coarse size air bubbles would also be increased then resulted in lower stability of air bubbles.

The air size distribution of mortar samples produced with the same mix-proportion but with different mixing time with AE is shown in **Fig. 1.27**.

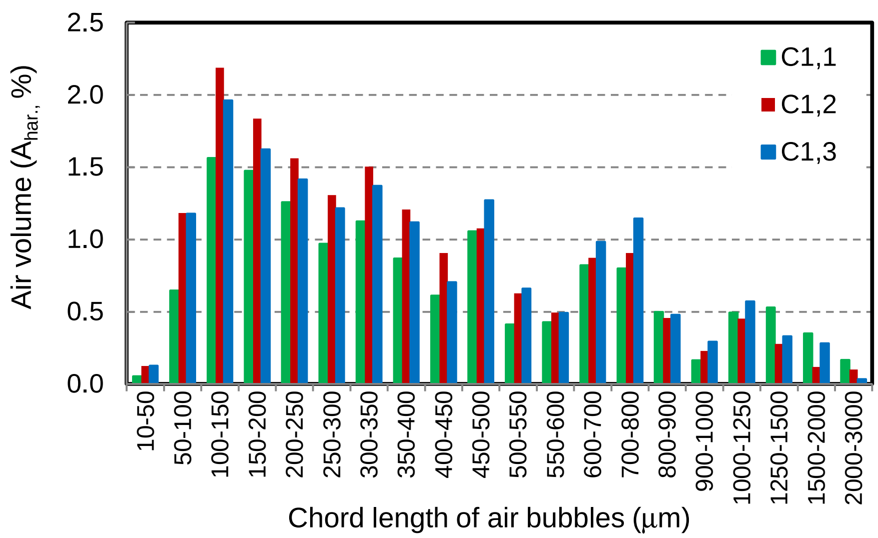



**Fig.1.27 Air bubbles distribution of mortar samples produced with different mixing time with AE (Y = 1 min, 2 min, or 3 min)**

Increasing mixing time with AE from 1 minute to 2 minutes increased mostly the content of air bubbles with the chord length of lower than 500 μm and decreased slightly the content of coarse size air bubbles. On the other hand, increasing mixing time with AE from 2 minutes to 3 minutes slightly lowered the content of small size air bubbles and increased the content of coarse size air bubbles. The correlation of share of air volume at coarse size and the increase in mixing time with AE is shown in **Fig. 1.28**. This result explained that the optimum mixing time with AE, the mixing time that could produce the finest air-voids system existed.

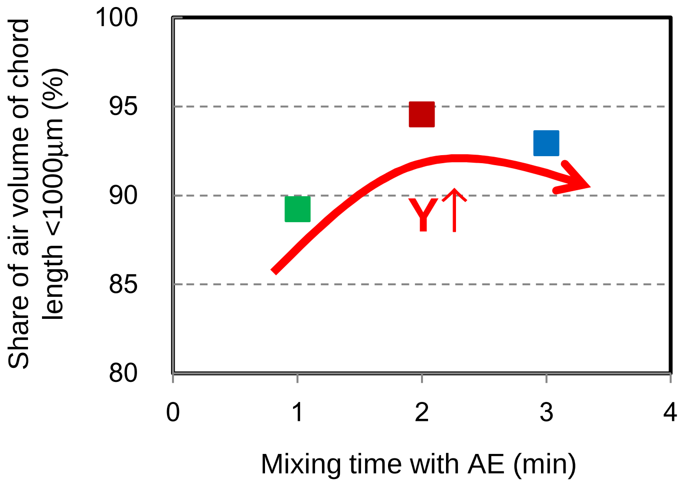



**Fig.1.28 Increasing the air volume of fine bubbles with longer mixing time with AE**

The effect of mixing time with SP on initial air content and stability of air bubble in 2 hours is shown in **Fig. 1.29**.

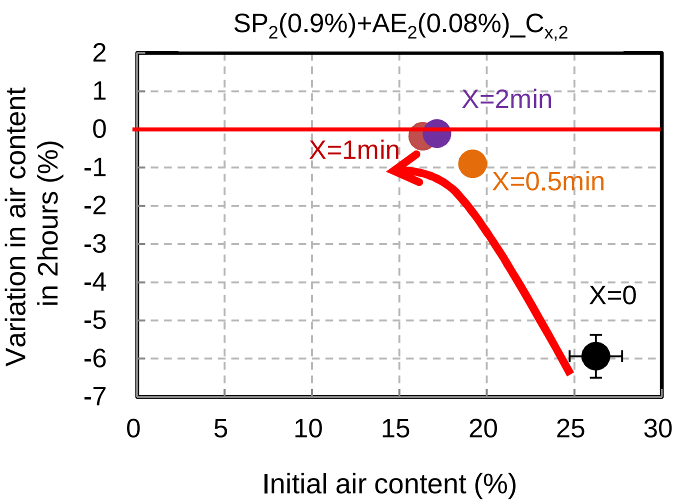



**Fig.1.29 Effect of mixing time with SP on initial air content and stability of air bubbles in 2 hours**

With longer mixing time with SP, initial air content reduced whereas the stability of air bubbles improved. Longer mixing time with SP provided more chance for the mixture to be dispersed before pouring AE thus lowering the viscosity of the mixture. As a result, lower content of air was obtained with higher amount of small size air bubbles. The small air bubbles itself were more stable than the larger ones. The stability of air bubbles improved by longer mixing time with SP.

The air size distribution of mortar samples produced with the same mix-proportion but with different mixing time with SP is shown in **Fig. 1.30**.

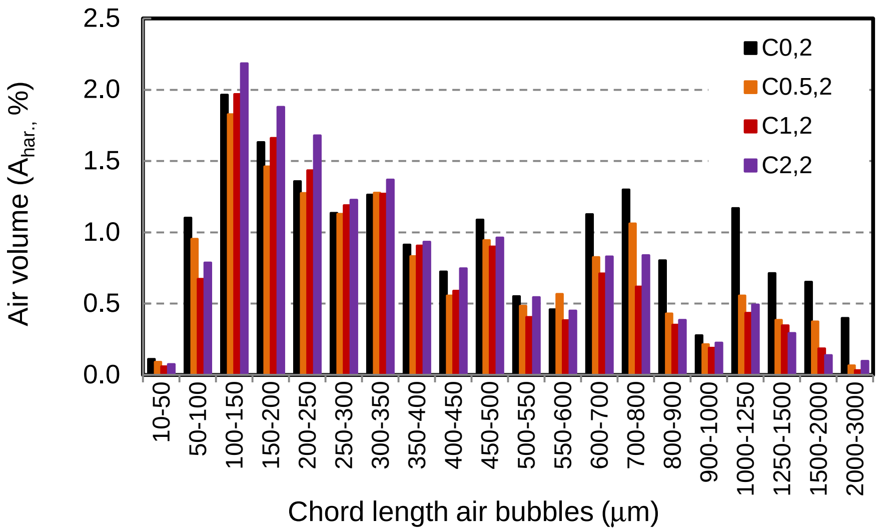



**Fig.1.30 Air size distribution of mortar samples produced with different mixing time with SP (X = 0 min, 0.5 min, 1 min, or 2 min)**

Air size distribution of the mortar samples in which SP was introduced before pouring AE ("X" ≠ 0) was different from that of the sample that SP and AE were introduced at the same time ("X" = 0). In case of the sample made with the mixing time with SP (before pouring AE) of 0 minute, high content of air with coarse size air bubbles was found. When the mixing time with SP was increased from 0 to 0.5 minute, a significant reduction in content of coarse size air bubbles could be obtained. Increasing the mixing time with SP from 0.5 minute to 1 minute then to 2 minutes increased the content of small size air bubbles (especially with chord length smaller than 250 μm) and gradually decreased the content of coarse size air bubbles. This result showed a significant improvement of pouring SP before pouring AE even with a very short mixing time. Also, longer mixing time with SP would improve the fineness of air-voids system to some high level.

The share of air volume at five ranges of chord length of mortar samples with the same mix-proportion produced with different mixing time with SP are shown in **Fig. 1.31.**

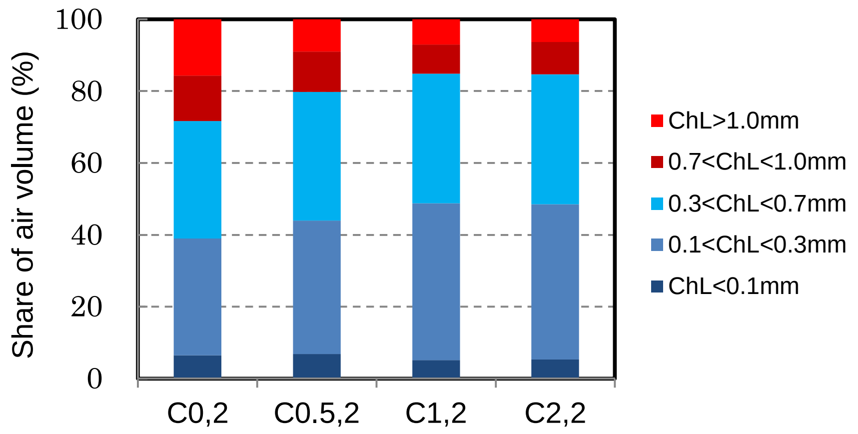



**Fig.1.31 Share of air volume of each range of chord length (ChL) of mortar samples produced with different mixing time with SP (X = 0 min, 0.5 min, 1 min, or 2 min)**

Longer mixing time with SP basically lowered the content of coarse size air bubbles. As a result, the share of volume of coarse size air bubbles was reduced. The share of air volume in mortars produced with mixing time with SP of 1 minute and 2 minutes were similar. This result showed that a maximum mixing time with SP which produced an optimum fineness of air entrainment existed. The reduction in air volume of coarse bubbles with longer mixing time with SP is shown in **Fig. 1.32**.

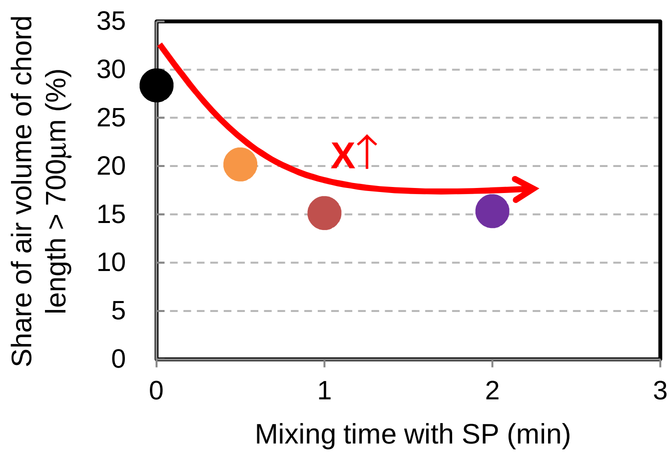



**Fig.1.32 Effect of mixing time with SP on minimizing the coarse air volume**

***e) Observation 3: Higher dosage of AE reduced the air volume of coarse bubbles***

Air size distribution of mortar measured with the linear traverse method and produced at different dosage of AE in case of mixing procedure $A_2$, $B_{1,1}$ or $C_{1,1}$ is shown respectively in **Fig. 1.33, Fig. 1.34** and **Fig. 1.35**.

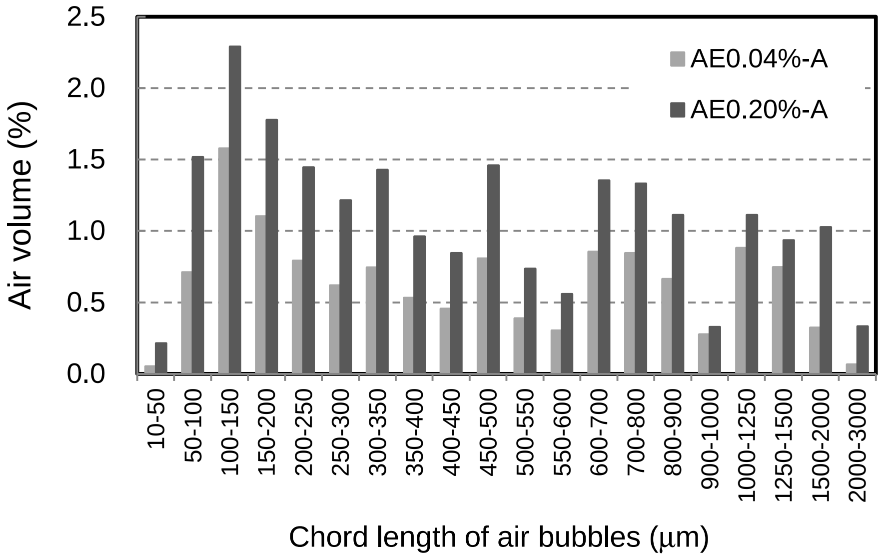



**Fig.1.33 Air size distribution affected by dosage of AE in case of mixing procedure A**

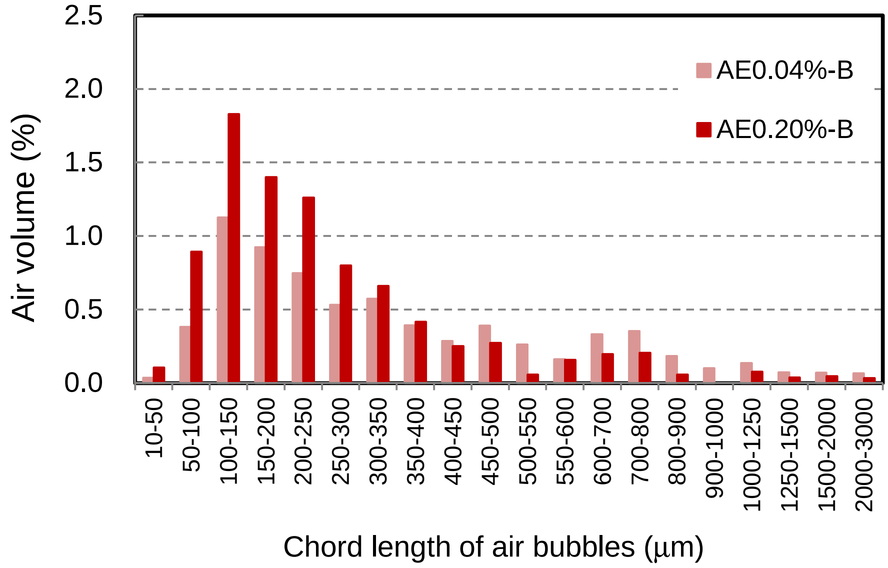



**Fig.1.34 Air size distribution affected by dosage of AE in case of mixing procedureB**

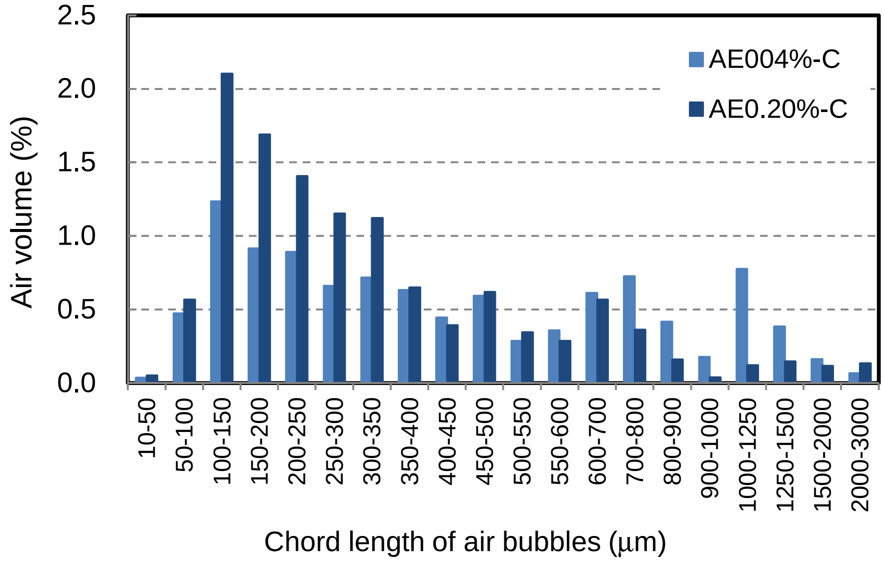



**Fig.1.35 Air size distribution affected by dosage of AE in case of mixing procedure C**

With mixing procedure A, higher dosage of AE increased air volume at each range of air bubble size thus causing no improvement in fineness of air entrainment. In case of mixing procedure B or C, higher dosage of AE increased air volume of fine size air bubbles while decreasing the volume of coarser size air bubbles. That phenomenon was beneficial to both fineness of air entrainment and the stability of air. Higher dosage of AE was required in case of mixing procedure B or C to reach same air volume as mixing procedure A. As it can be seen in **Table 1.7**, even at a low dosage of AE, air volume produced with mixing procedure A was highly larger than mixing procedure B or C. Such a low dosage of AE caused poor stability of air due to high volume of air at coarse size bubbles.

The relationship between dosage of AE and the share of the volume of coarse size air bubbles is shown in **Fig. 1.36**.

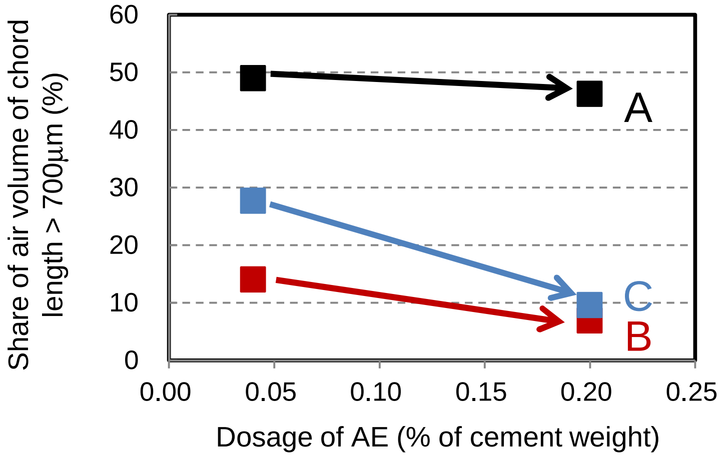



**Fig.1.36 Reduction in share of coarse air volume by the increase in dosage of AE in the case of effective mixing procedure**

It can be seen that the increasing dosage of AE improved the fineness of air distribution through reducing the volume of coarse size air bubbles. Interestingly, with the mixing procedure A, there was a significant reduction in coarse air volume when the dosage of AE was increased while the share of coarse air volume was high comparing to that of the mixing procedure B or C.

## 1.7. Purpose of Research

The purpose of this research is to modify the mixing procedure to effectively improve the stability of air bubbles and to be able to reach target air volume with satisfied fineness of air entrainment. A controllable air entrainment is necessary for a production of air-SCC with adequate stability of air bubbles and insurable self-compactability. To achieve stable air entrainment, the target of this research is to minimize the air volume of coarse bubbles as shown in **Fig. 1.37**.

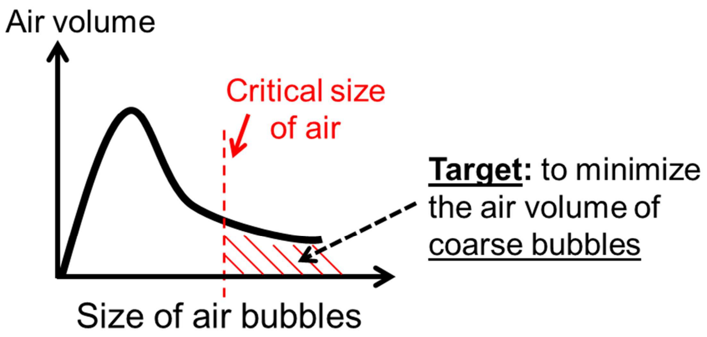



**Fig.1.37 Purpose of the research: How to minimize the air volume of coarse bubbles**

## 1.8. Outline of Research

To initiate the effective mixing procedure which is able to ensure the stability of air and to reach the target air volume with satisfied fineness of air bubbles distribution, the outline of this research is as following. Since the difference in the mixing procedure and its effectiveness on the stability of air as much as the air bubbles distribution were introduced already in this chapter, the following chapters are to determine firstly a more precise critical air bubble size to the stability of air by using the air bubbles distribution measuring at fresh stage. After this critical size can be determined, it is able to distinguish the favorable size of air bubbles (fine air) and the unfavorable size of air bubbles (coarse air). With a clear distinction, the clarification of the viscosity due to different type of mixing procedure on entraining air volume of coarse or fine bubbles was conducted through varying water to cement ratio. Then, the entraining air volume of coarse or fine air bubbles due to different dosage of AE was clarified. Furthermore, the capacity of the mixing time in reaching the upper limit volume of fine air will also be clarified. This upper limit was expected to be defined by the combination between the viscosity due to different mixing procedure and the dosage of AE.

The clarifications mentioned above are for proposing a method for entraining stable air is introduced through adjusting the mixing time with AE and the dosage of AE considering the viscosity of the mixture. The brief outline of this research is shown in **Fig. 1.38**.

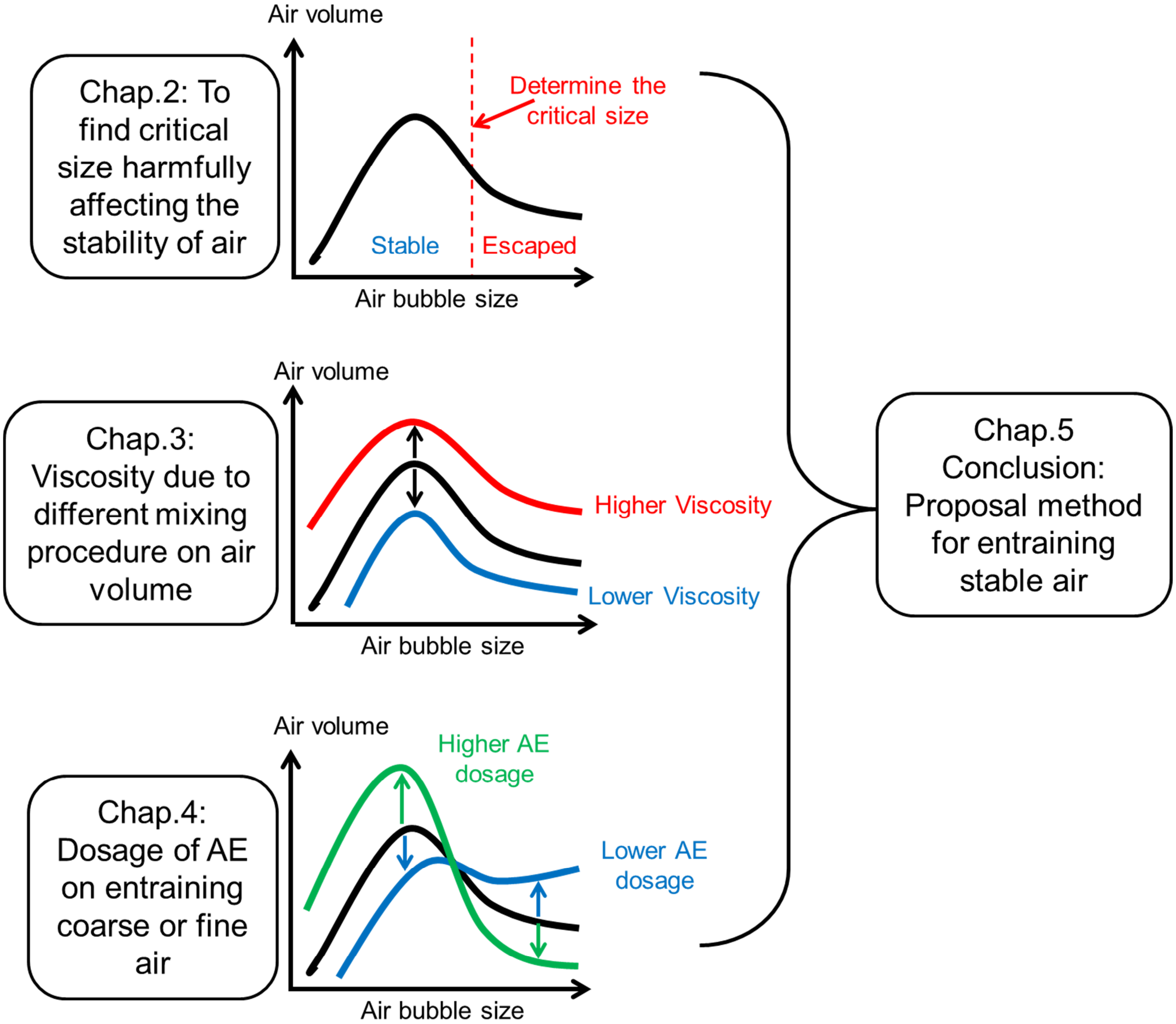



**Fig.1.38 Outline of research**

# CHAPTER 2 CRITICAL SIZE OF ENTRAINED AIR TO STABILITY OF AIR VOLUME AT FRESH STAGE

## 2.1. Purpose

The purpose of this chapter is to determine the critical size of air bubbles which may harmfully affect the stability of air. Different type of mixing procedure was modified aiming to improve the stability of air. After the critical size was determined, the choice of effective mixing procedure and its adjustment can be made. Once the air volume of coarse bubbles is able to minimized, the stability of air would be improved and the target air volume would be adjustable.

## 2.2. Hypothesis

A coarse air bubbles floats faster than the smaller one since due to smaller surface friction of air. The coarse air bubble which is critical to the stability of air is defined as those that escapes easily either by collapsing in the matrix or by floating upward then fading away. Air bubble with smaller size (higher internal pressure) is more stable than coarser size air bubble (lower internal pressure). The mixture containing high volume of coarse size air bubbles would be more likely to possess a poor stability of air. The definition on the critical defined for this study is shown in **Fig. 2.1**.

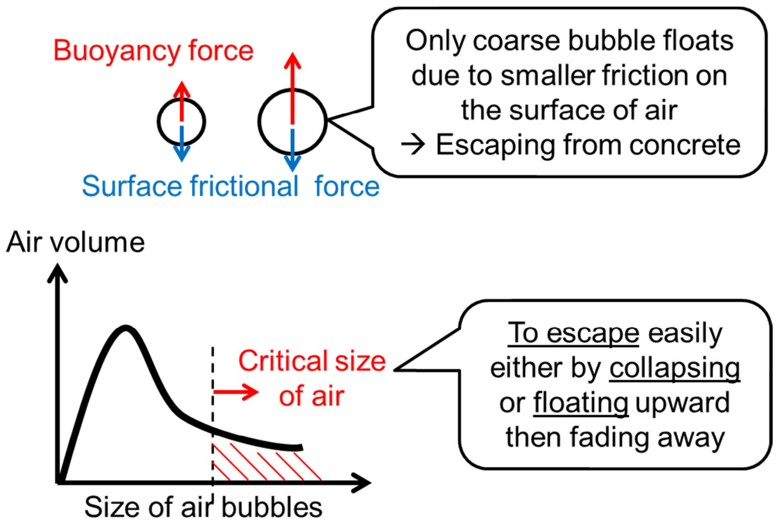



**Fig.2.1 Definition of the critical size of air bubbles**

### 2.3. Determination of Critical Size of Air Bubbles to the Stability of Air

#### 2.3.1. Mortar samples and mixing procedure

To determine the critical size of air bubbles, air size distribution of mortar samples were measured at fresh stage with air-voids analyzer (AVA) test method. 30 mortar samples were made with different types of mixing procedure and with variation on mixing time with AE and dosage of AE. The mixing procedure used in this chapter is shown in **Fig. 2.2**. The mix-proportion and properties of these mortar samples are shown in **Table 2.1**.

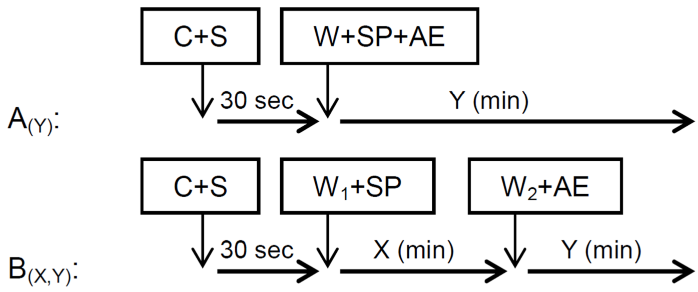



**Fig.2.2 Mixing procedure for mortar to be tested air distribution with AVA test method**

**Table 2.1 Mix-proportion and properties of mortar samples produced with mixing procedure A and tested with AVA-3000**

| N° | Mixing procedure | $SP_1/C$ (%) | $AE_1/C$ (%) | Flow (mm) | Funnel speed | Air content (%) | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | $A_{initial}$ | $A_{2h}$ | $A_{>500\mu m}$(*) | $A_{<500\mu m}$ |
| 1 | $A_2$ | 1.4 | 0.005 | 247 | 2.48 | 11.1 | 7.5 | 8.8 | 2.3 |
| 2 | $A_6$ | 1.2 | 0.005 | 260 | 3.73 | 13.5 | 6.0 | 11.8 | 1.7 |
| 3 | $A_2$ | 1.4 | 0.010 | 252 | 2.65 | 13.0 | 10.5 | 6.6 | 6.4 |
| 4 | $A_4$ | 1.2 | 0.010 | 256 | 3.24 | 18.0 | 13.4 | 10.9 | 7.1 |
| 5 | $A_6$ | 1.2 | 0.010 | 251 | 3.55 | 21.6 | 14.5 | 15.3 | 6.3 |
| 6 | $A_2$ | 1.4 | 0.020 | 245 | 2.46 | 18.3 | 17.5 | 4.1 | 14.2 |
| 7 | $A_4$ | 1.2 | 0.020 | 254 | 2.89 | 22.8 | 22.2 | 3.4 | 19.4 |
| 8 | $A_6$ | 1.2 | 0.020 | 258 | 3.09 | 24.0 | 22.9 | 2.2 | 21.8 |
| 9 | $A_8$ | 1.2 | 0.020 | 262 | 3.38 | 20.3 | 19.3 | 5.4 | 14.9 |
| 10 | $A_2$ | 0.7 | 0.005 | 196 | 2.10 | 11.6 | 7.6 | 10.1 | 1.5 |
| 11 | $A_2$ | 0.7 | 0.010 | 193 | 2.19 | 15.8 | 10.3 | 12.5 | 3.3 |

**Table 2.2 Mix-proportion and properties of mortar samples produced with mixing procedure B and tested with AVA-3000**

| N° | Mixing procedure | $SP_1/C$ (%) | $AE_1/C$ (%) | Flow (mm) | Funnel speed | Air content (%) | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | $A_{initial}$ | $A_{2h}$ | $A_{>500\mu m}$(*) | $A_{<500\mu m}$ |
| 1 | $B_{1,2}$ | 1.1 | 0.010 | 236 | 3.32 | 6.4 | 6.9 | 3.8 | 2.6 |
| 2 | $B_{1,4}$ | 0.9 | 0.010 | 227 | 3.73 | 6.8 | 7.2 | 2.3 | 4.5 |
| 3 | $B_{1,6}$ | 0.9 | 0.010 | 237 | 3.88 | 7.1 | 7.0 | 2.3 | 4.8 |
| 4 | $B_{1,10}$ | 0.9 | 0.015 | 229 | 3.94 | 8.1 | 8.2 | 1.6 | 6.5 |
| 5 | $B_{1,15}$ | 0.9 | 0.015 | 223 | 3.91 | 11.3 | 11.1 | .6 | 9.7 |
| 6 | $B_{1,20}$ | 0.9 | 0.015 | 220 | 3.75 | 16.5 | 16.6 | 3.7 | 12.9 |
| 7 | $B_{1,2}$ | 1.1 | 0.020 | 262 | 4.07 | 6.2 | 6.7 | 1.8 | 4.4 |
| 8 | $B_{1,4}$ | 0.9 | 0.020 | 240 | 3.82 | 8.4 | 8.5 | 1.8 | 6.7 |
| 9 | $B_{1,6}$ | 0.9 | 0.020 | 242 | 3.83 | 9.2 | 9.6 | 1.4 | 7.8 |
| 10 | $B_{1,10}$ | 0.9 | 0.020 | 234 | 3.83 | 10.5 | 11.0 | 2.0 | 8.4 |
| 11 | $B_{1,15}$ | 0.9 | 0.020 | 227 | 3.91 | 11.9 | 12.1 | 1.1 | 10.8 |
| 12 | $B_{1,20}$ | 0.9 | 0.020 | 199 | 3.39 | 21.3 | 21.3 | 2.5 | 18.7 |
| 13 | $B_{1,25}$ | 0.9 | 0.020 | 209 | 3.39 | 19.3 | 18.5 | 2.2 | 17.1 |
| 14 | $B_{1,4}$ | 0.9 | 0.040 | 240 | 3.98 | 9.4 | 10.7 | 1.5 | 7.9 |
| 15 | $B_{1,6}$ | 0.9 | 0.040 | 241 | 4.20 | 11.2 | 11.3 | 1.2 | 10.0 |
| 16 | $B_{1,8}$ | 0.9 | 0.040 | 238 | 3.95 | 12.2 | 12.6 | 1.1 | 11.1 |
| 17 | $B_{1,6}$ | 0.9 | 0.080 | 230 | 4.00 | 13.4 | 13.8 | 2.2 | 11.2 |
| 18 | $B_{1,8}$ | 0.9 | 0.080 | 242 | 4.20 | 14.2 | 14.7 | 1.2 | 13.0 |
| 19 | $B_{1,10}$ | 0.9 | 0.080 | 234 | 4.10 | 15.6 | 15.8 | 0.4 | 15.2 |

*(*) The value of air volume of bubbles with chord length of larger than 500 $\mu$m was the results of the adjustment with the gap between air volume measured by weight and that measured by AVA, as explained in section* ***2.3.3****.*

### 2.3.2. Air Void Analyzer (AVA-3000) for measuring diameter of air in fresh mortar

#### a) Significance of measuring method

The AVA test method was developed to measure of air-void system of concrete or mortar at fresh stage. Before this invention, it is hard to fully understand the initial stage of air-void system which is required to assure that the target air entrainment is achieved. For this study, this test method is beneficial to fully understand the characteristics of the air-void system at fresh stage to be able to adjust the suitable mixing method and mix-proportion which encourage a finer system of air-voids and improve the stability of air bubbles.

#### b) Mechanism of measurement

The mechanism of AVA method is to expel all air bubbles included in a given mortar/concrete sample, collect the air bubbles, and record their quantities and size distribution. The sample is placed in the viscous release liquid by stirring using a magnet to release all air bubbles. With careful control on viscosity of the liquid, the air bubbles could retain their original size without neither coalesce nor disintegrate into smaller bubbles. According to the Stokes law, the floating speed of each air void rising through the liquid depends on its size. The viscosity of the analysis liquid slows down the initial rise of the bubbles providing a measurable separation in time to the arrival at the top of the column of bubbles of different sizes.

From this data, air void parameters calculated includes the air content, specific surface and spacing factors. These parameters are calculated to correspond to those that would be obtained from linear traverse measurements on a planar surface of the hardened concrete using the assumptions outlined in ASTM C 457: (1) the average measured chord length is equal 2/3 of the true air void diameter as shown in **Fig. 2.3** and (2) for the calculation of specific surface and spacing factor, the voids are all of the same size and they are located in lattice points of a regular cubic array.

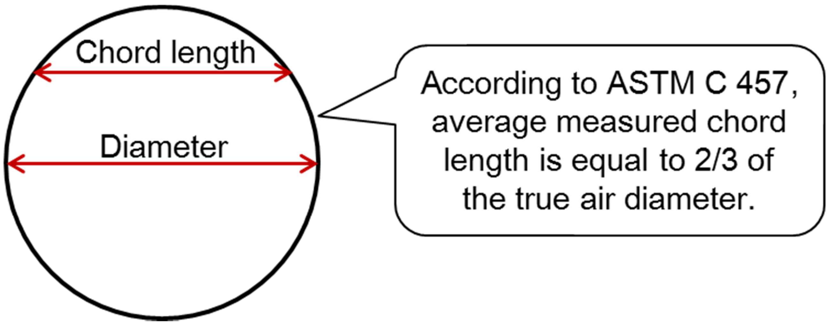



**Fig. 2.3 Assumption on the relationship between diameter and chord length of air bubble**

First of all, de-aerate tap water and temper the releasing liquids in the water tank at least 24 hours prior to testing air distribution. After one day, place the AVA base unit on top of the water tank and connect it by means of the connecting device to a computer having AVA application. Then place the riser column connected with mortar sample and piston as seen in **Fig. 2.4(a)** on the top of the AVA base unit and fix it with the screws. After that, place the magnetic stirrer rod in the riser column's bottom. Fill the water from the tank to the riser column to a level of about 3 cm below its top edge and carefully remove all bubbles in the riser column by the brush. Fill the funnel with releasing liquid and then gently place it into the riser column. Finally, place the buoyancy pan on the balance then position the windshield on top of the riser column.

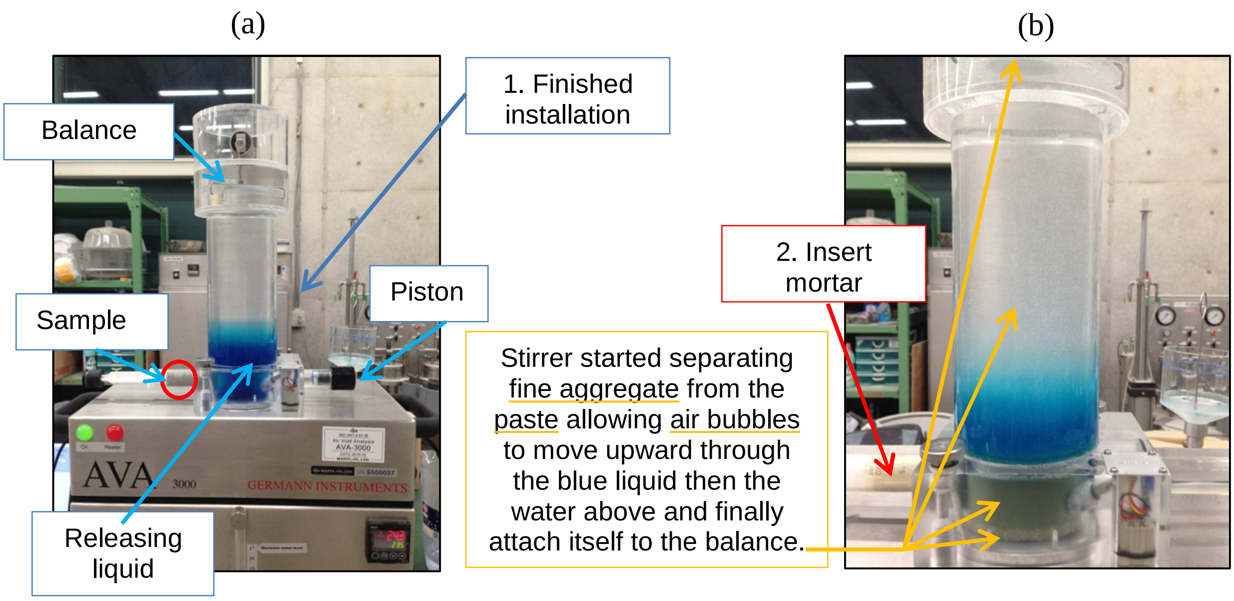



**Fig.2.4 (a): Equipment of AVA-3000, (b): Rising bubbles during measurement**

After finishing installation, gently remove the piston and insert the mortar sample into the riser column. Then start the AVA testing program. The magnetic stirrer will separate the fine aggregate from the cement paste and then the releasing liquid releases the air bubbles allowing bubbles to rise through the riser column and attach itself to the buoyancy pan. The rising of bubbles through riser column is shown in **Fig. 2.4(b)**.

Examples of AVA-3000 result printed out from the measurement are shown in **Fig. 2.5** and **Fig. 2.6** respectively. In **Fig. 2.5**, the horizontal axis shows the change in mass of the buoyancy pan as a function of time (vertical axis). The results of the analyses include the air content in concrete, air content in paste, air content in putty, specific surface, and spacing factor. The chord length of 2mm and 1mm corresponds to air void diameter of 3mm and 1.5mm respectively, according to ASTM C 457.

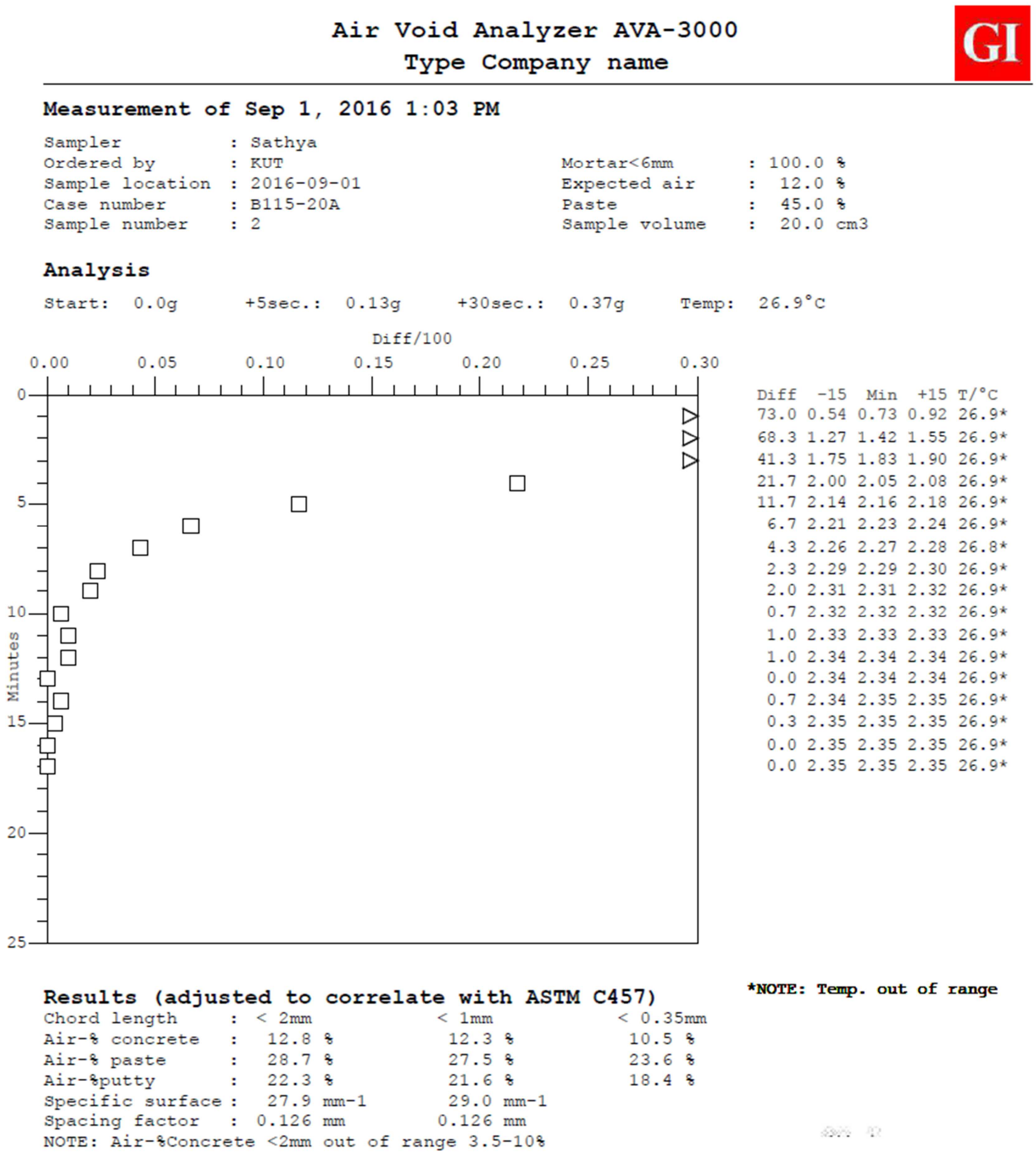



**Fig.2.5 Example of result from measurement with air void analyzer (AVA-3000)**

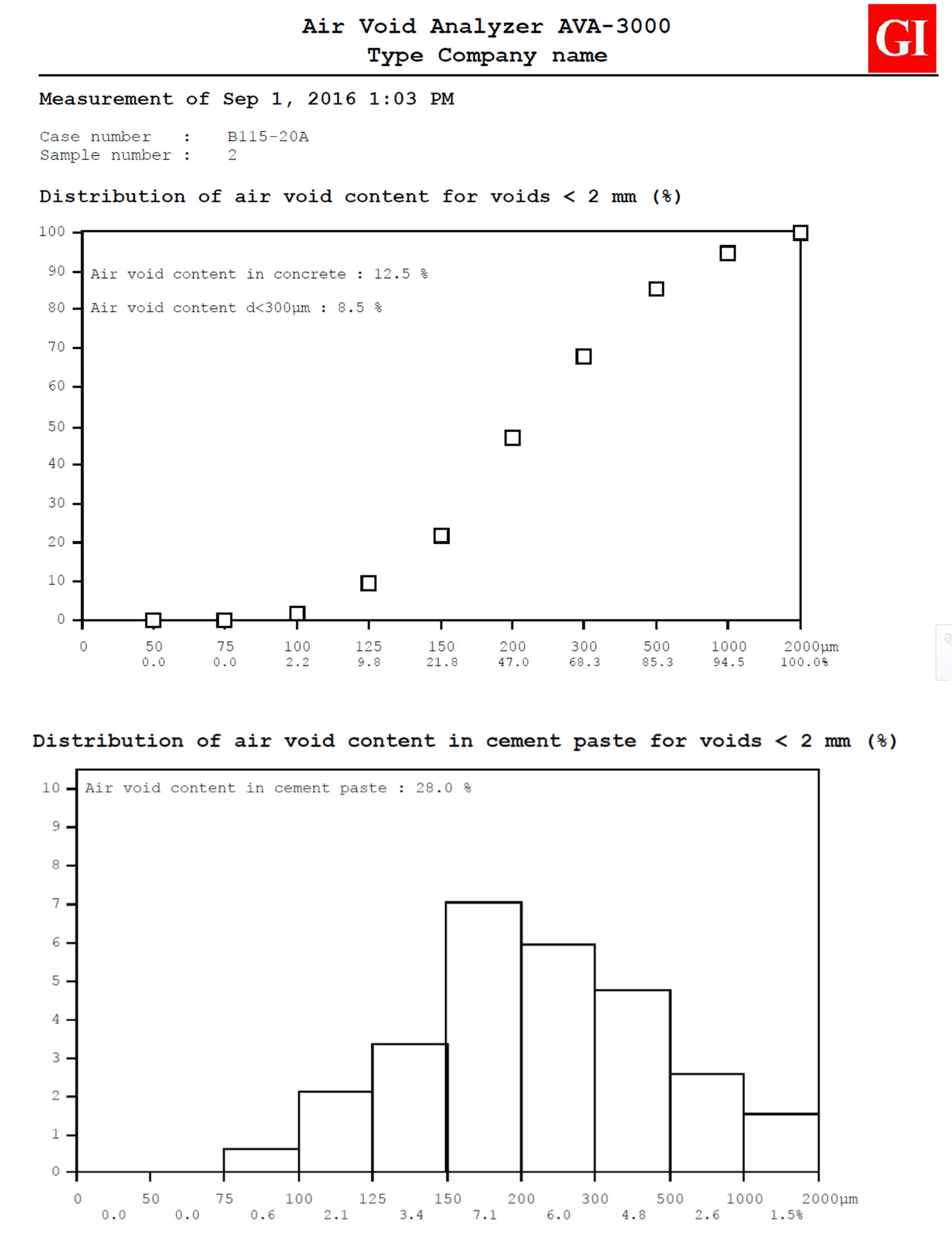



**Fig.2.6 Example of result of distribution of air bubbles measured with AVA-3000**

### 2.3.3. Determination of critical size of air bubbles to the stability of air

***a) Determination directly from AVA***

The relationship between the stability of air and the air content at fresh stage of each chord length range (>100 μm, >300 μm, >500 μm, or >1000 μm) are shown respectively in **Fig. 2.7**.

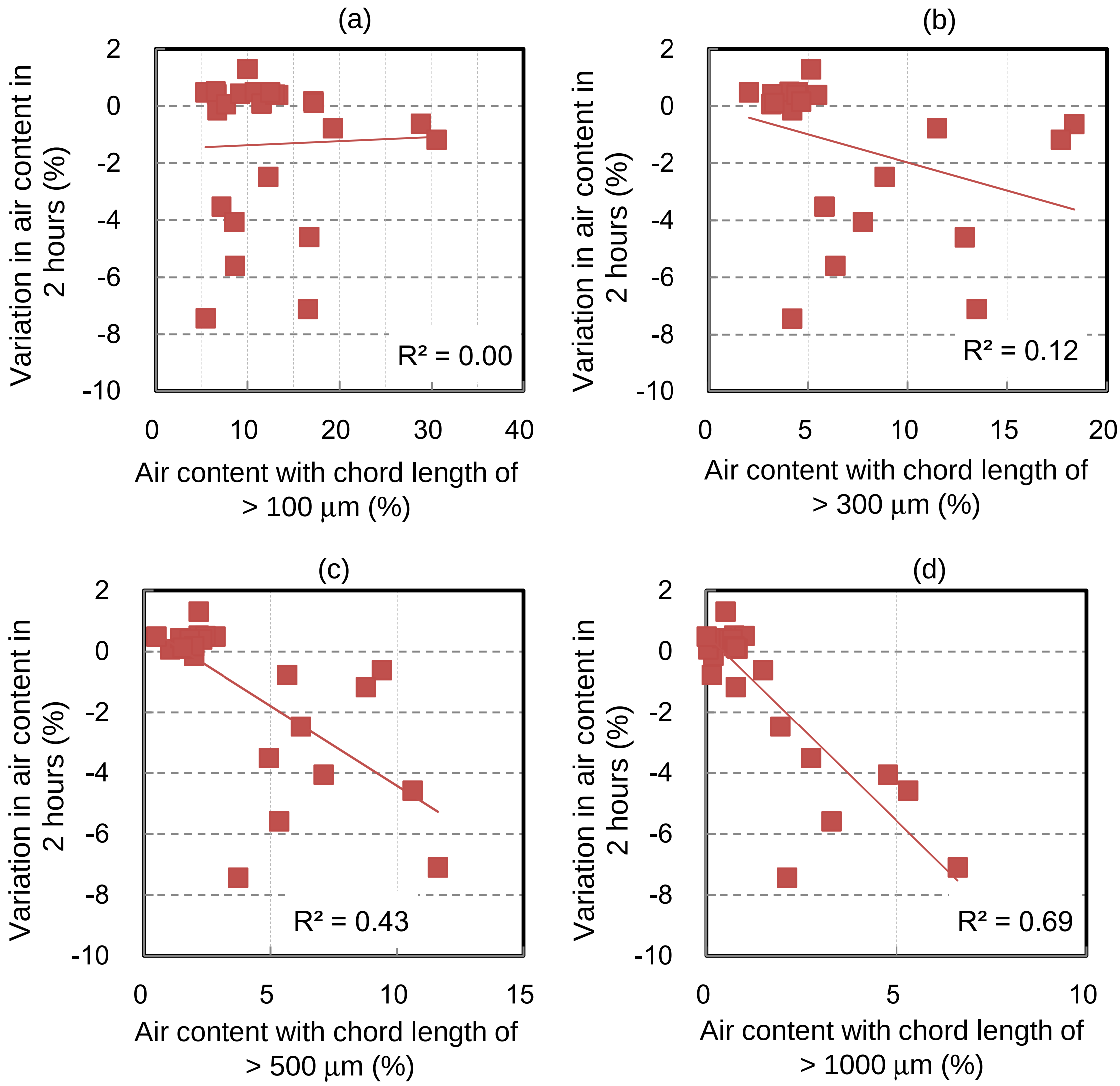



**Fig.2.7 Relationship between the stability of air in 2 hours and the air content at fresh stage with chord length of (a): >100 μm, (b): >300 μm, (c): >500 μm, or (d): >1000 μm**

Correlation between the stability of air and the air content at fresh stage at each chord length is shown in **Fig. 2.8**.

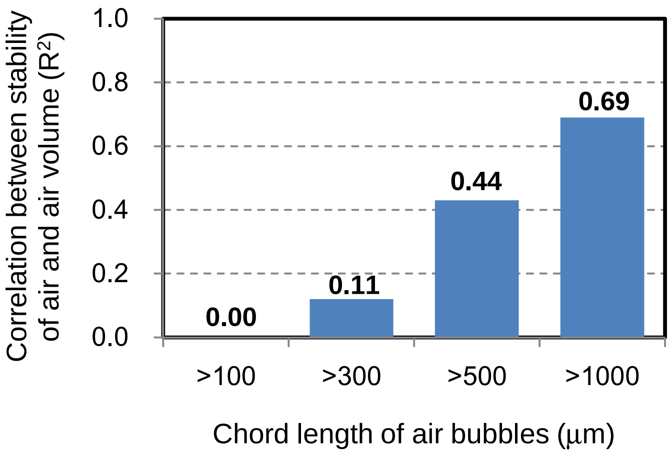



**Fig.2.8 Correlation between the stability of air in 2 hours and air content at fresh stage (measured with AVA) of each range of chord length**

It can be seen that the stability of air is more related to the content of coarser air bubbles than the smaller ones. The highest correlation ($R^2$) of 0.69 was obtained with the chord length of larger than 1000 µm. This result showed that the stability of air influenced mainly by the air volume of bubbles with chord length larger than 1000 µm.

***b) Consideration of escaping air during sampling***

The measurement of air volume was done with gravitic method prior to sampling process for air void distribution measuring with also AVA machine. The correlation between air volume measured with weight and the air volume measured with AVA is shown in **Fig. 2.9**.

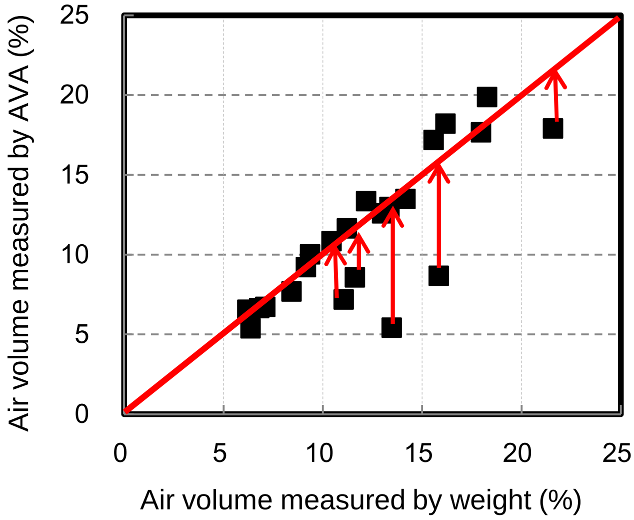



**Fig.2.9 Correlation between air volume measured with weight and with AVA**

There were some cases where the measurement of air volume with AVA was considerably lower than that measured by weight. This explained that there were cases where the air-entrained mortar sample of poor stability could easily cause a reduction in air volume during sampling the sample for AVA test. That air volume may have belonged to the coarse size air bubbles as it escaped faster than that of the smaller ones and will be more critical to be lost if there were large volume of air of that coarse size.

To improve the reliability of the determination on the critical size of air bubbles to the stability of air, adjustment on the total coarse air volume were made as shown in **Fig. 2.9**.

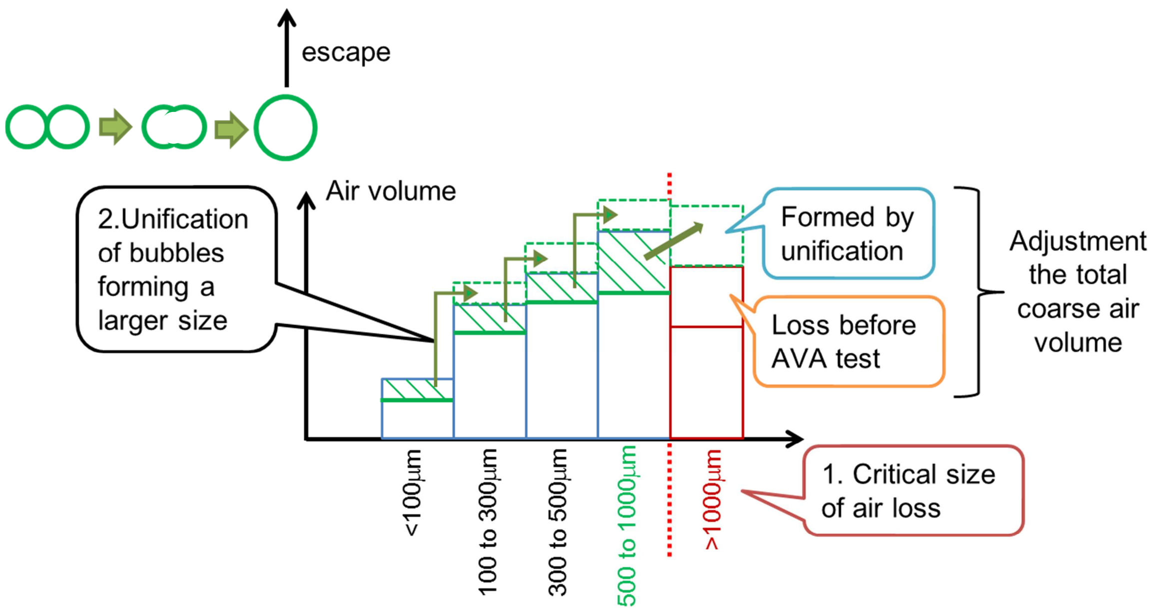



**Fig.2.10 Adjustment of total coarse air volume considering air loss during sampling and unification between air bubbles**

In this assumption, firstly the air bubbles of chord length larger than 1000 μm was considered as the critical value to the air loss based on the highest correlation resulted in **Fig. 2.8**. Thus, the difference between air volume measured by weight and that measured with AVA was as the coarse air with chord length of larger than 1000 μm which were lost during the sampling of mortar.

***c) Consideration of unification between air bubbles***

Since air entrainment changed when time passed, there would be unification between air bubbles occurred before the experiment was done in 2 hours. Thus, by considering that during that time small air bubbles would be unified together forming larger size ones, part of the air volume with chord length range 500 μm to 1000 μm was also unified to form bubbles of chord length larger than 1000 μm. An example on the variation in air size distribution from initial stage to 2 hours is in shown in **Fig. 2.11**.

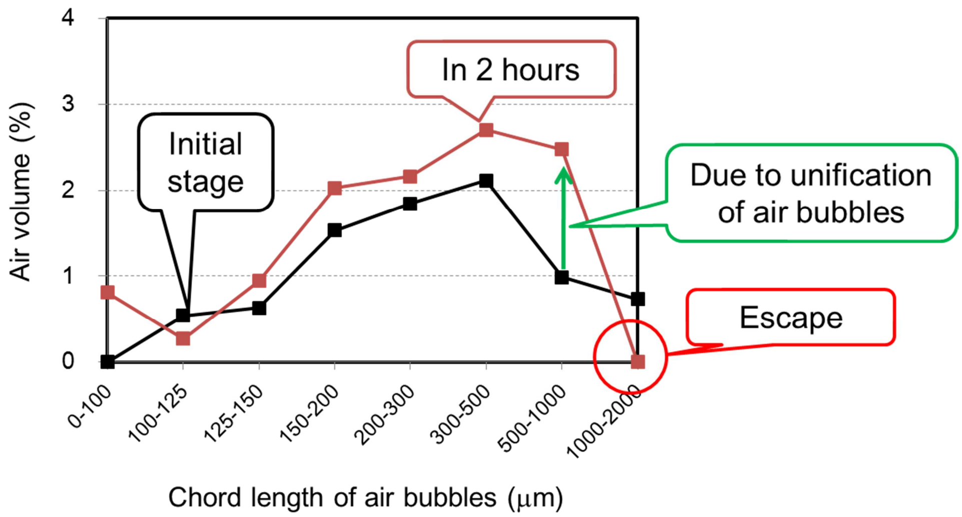



**Fig. 2.11 Example of variation in air size distribution in 2 hours measured with AVA**

***d) Critical size of air bubbles to the stability of air***

Following the adjustment process mentioned above, the correlation between air volume of bubbles with chord length of larger than 1000 μm, 500 μm, 300 μm or 100 μm to the stability of air is shown in **Fig. 2.12**.

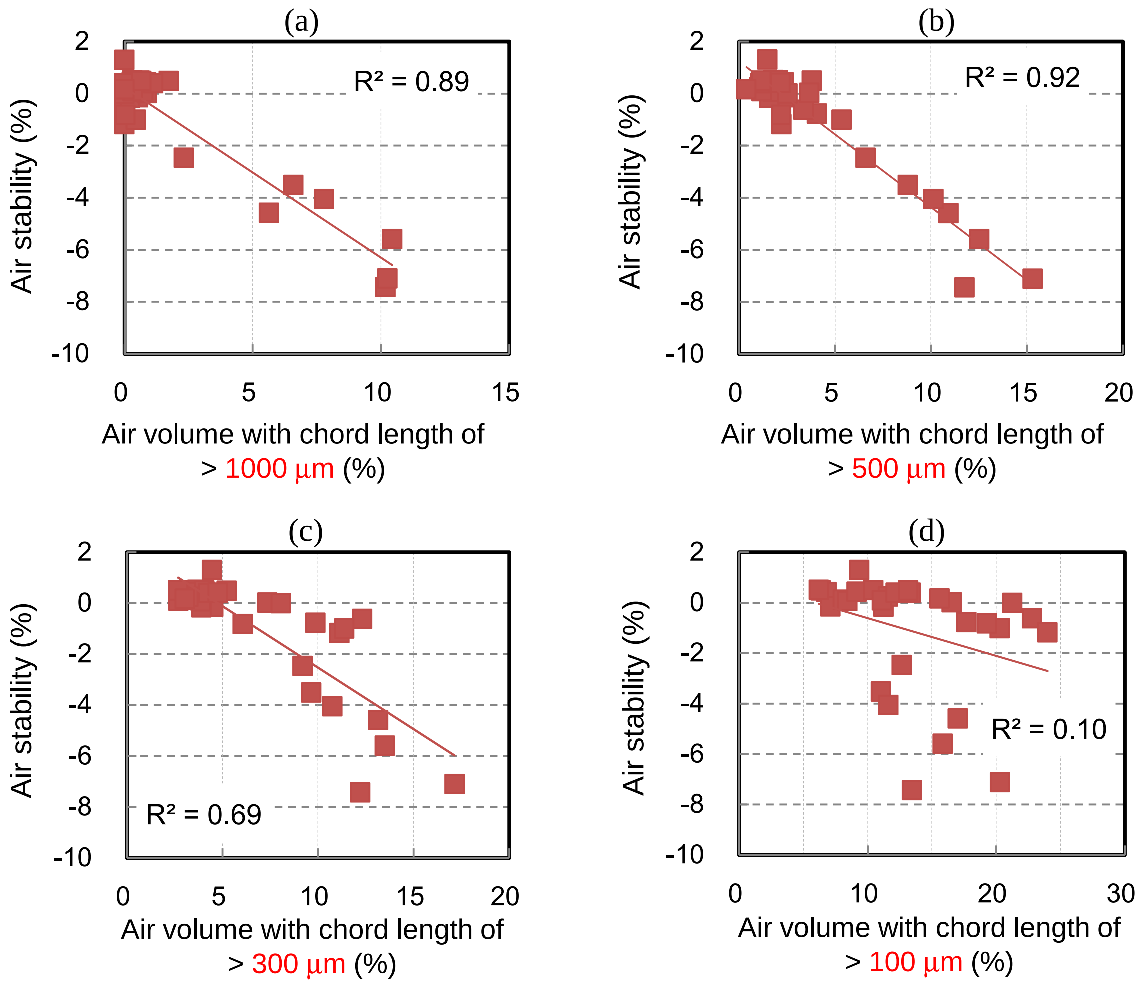



**Fig.2.12 Correlation between the stability of air to the air volume after adjustment with chord length of > 1000 µm, > 500 µm, > 300 µm or > 100 µm**

To precisely consider the unification of air bubbles, different partially share of air volume with chord length of 500 μm to 1000 μm including to that of chord length larger than 1000μm was analyzed to correlate with the stability of air as shown in **Fig. 2.13**.

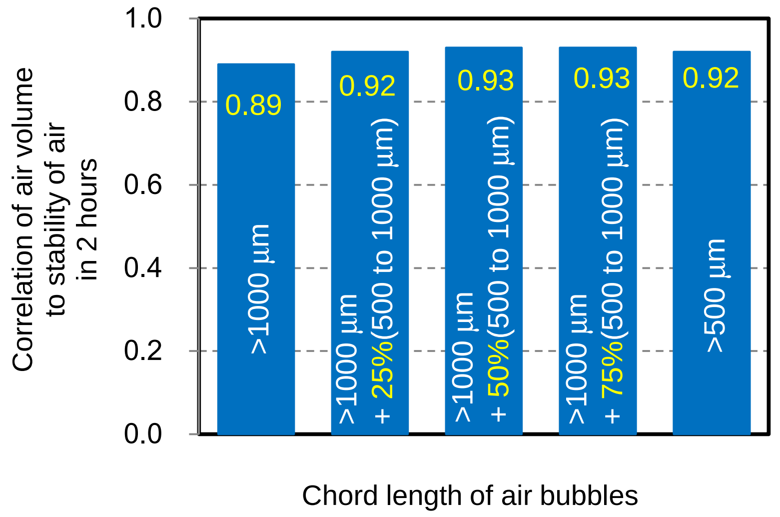



**Fig.2.13 Correlation between air volume to the stability of air considering the unification of air at different portion**

It can be seen that by including 50% or 75% of air volume with chord length of 500 μm to 1000 μm to the volume of coarse air with chord length of larger than 1000 μm, the highest correlation of 0.93 was obtained. These correlations are shown in **Fig. 2.14**.

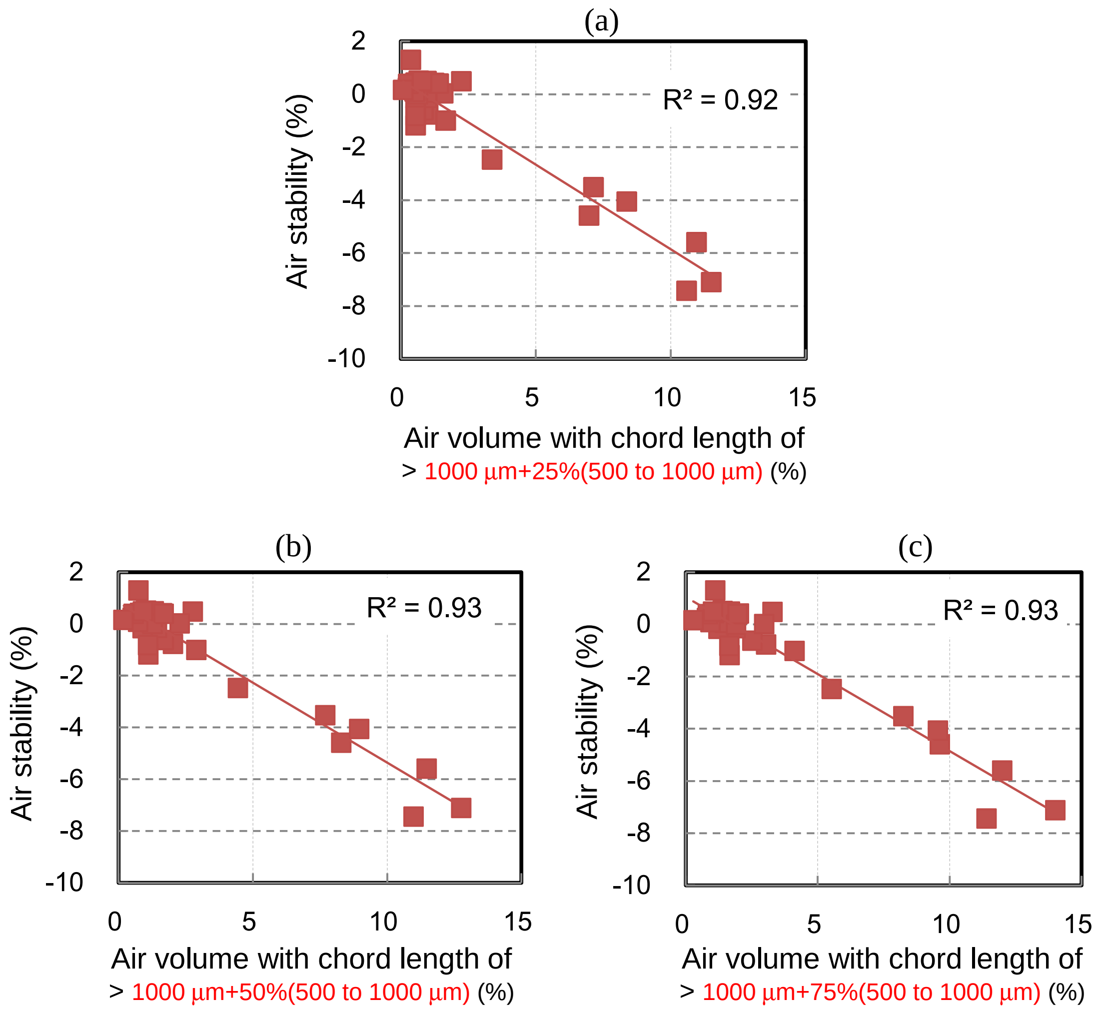



**Fig.2.14 Correlation between the stability of air and the volume with chord length of larger than 1000 μm + 25%, 50% or 75% of unification of air volume with chord length of 500 μm to 1000 μm**

It is possible that the existence of air volume with chord length of larger than 1000 μm and the partial air volume with chord length of 500 μm to 1000 μm due to the unification caused the instability of air in fresh mortar of self-compacting concrete. However, to simplify the further analysis in the next chapter, the coarse air bubbles which was harmful to the stability of air was chosen to be with the chord length of over 500 μm. Thus, to ensure the stability of air means to minimize the air volume at this range.

## 2.4. Critical Size of Air Bubbles to the Stability of Air in Concrete

### 2.4.1. Experimental plan

This section is the clarification in case of concrete whether the critical size of air bubbles determined in the case of mortar was also applicable for concrete or not. At concrete plant, to verify the critical size of air bubbles to the stability of air, four samples of concrete were conducted with mixing procedure $A_{(Y)}$. The properties of materials used for concrete experiments in this section are shown in **Table 2.2**. The mix-proportion and the properties of these samples at initial stage are shown in **Table 2.3**.

**Table 2.3 Materials used for concrete experiments**

| | |
|---|---|
| Cement (C) | Ordinary Portland cement (3.15g/cm$^3$) |
| Fine aggregate (S) | Crushed limestone sand (2.68g/cm$^3$, F. M. 2.96) |
| Superplasticizer (SP) | $SP_1$: Polycarboxylic based with viscosity agent |
| | $SP_3$: Polycarboxylic based with viscosity agent and retarding type |
| | $SP_4$: Polycarboxylic based |
| Air-entraining agent (AE) | $AE_1$: Alkyl ether-based anionic surfactants *(AE: water concentration is 1:99)* |

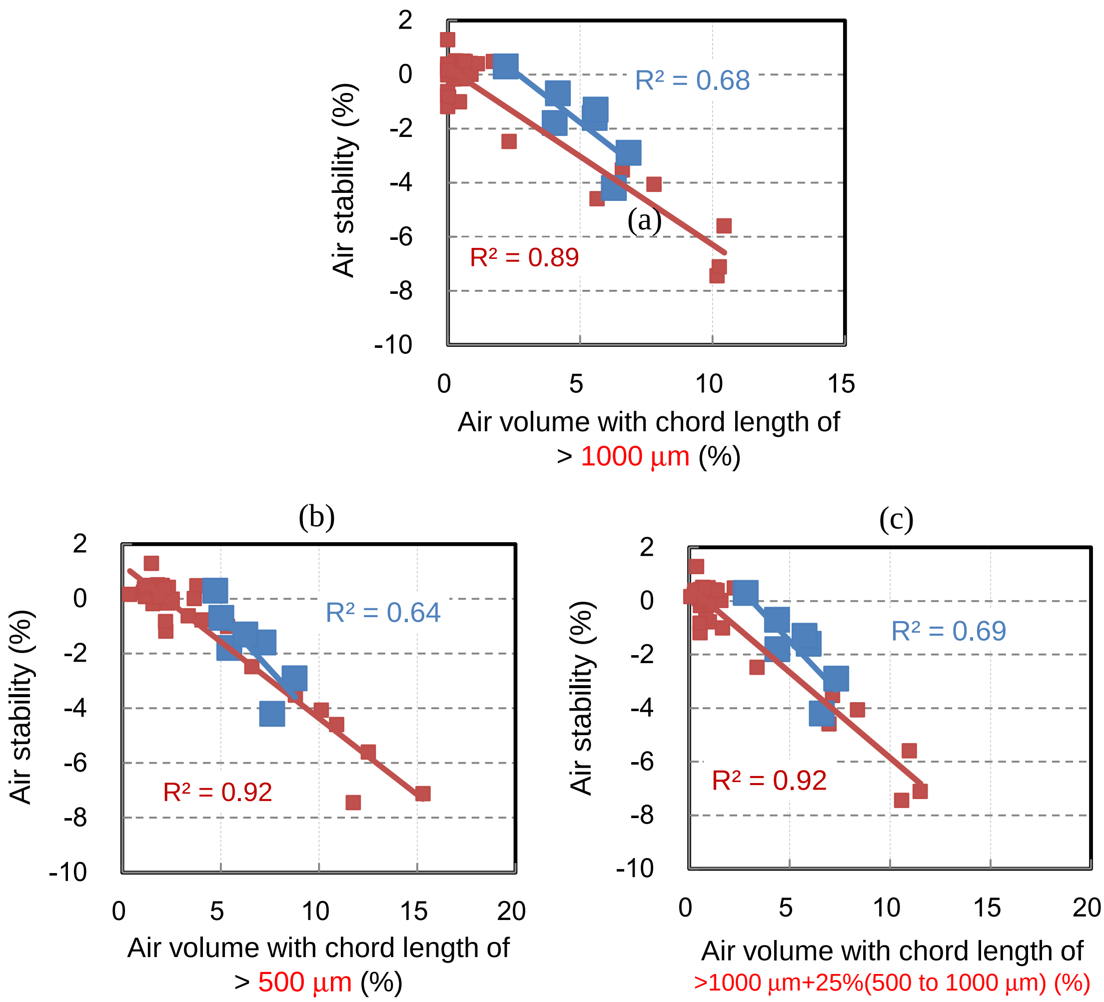
**Table 2.4 Mix-proportion of each concrete sample**

| N° | Mixing procedure | SP/C | | AE/C | | Properties at initial stage | | |
|---|---|---|---|---|---|---|---|---|
| | | Type | (%) | Type | (%) | Flow (mm) | Funnel speed | Air content (%) |
| 1 | $B_{1.5,1.5}$ | $SP_1$ | 1.1 | $AE_1$ | 0.015 | 595 | 2.04 | 9.7 |
| 2 | $A_{1.5}$ | $SP_1$ | 1.1 | $AE_1$ | 0.015 | 570 | 1.47 | 10.5 |
| 3 | $A_3$ | $SP_3$ | 1.0 | $AE_1$ | 0.015 | 518 | 1.69 | 10.7 |
| 4 | $A_3$ | $SP_3$ | 1.2 | $AE_1$ | 0.015 | 578 | 1.64 | 11.5 |
| 5 | $A_{3,1(3T)}$* | $SP_3$ | 1.1 | $AE_1$ | 0.015 | 630 | 1.98 | 7.6 |
| 6 | $A_{3,1(6T)}$ * | $SP_3$ | 1.1 | $AE_1$ | 0.015 | 650 | 1.94 | 7.9 |
| 7 | $A_{1.5,1(3T)}$* | $SP_3$+$SP_4$ | 0.75+0.75 | $AE_1$ | 0.015 | 630 | 1.85 | 7.9 |

(*) 1 minute mixing with de-foaming agent

### 2.4.2. Results

The correlation between air volume with bubble size of larger than 1000 μm plus about 50% of bubbles with chord length between 500 μm to 1000 μm and the stability of air in concrete is shown in **Fig. 2.15**.



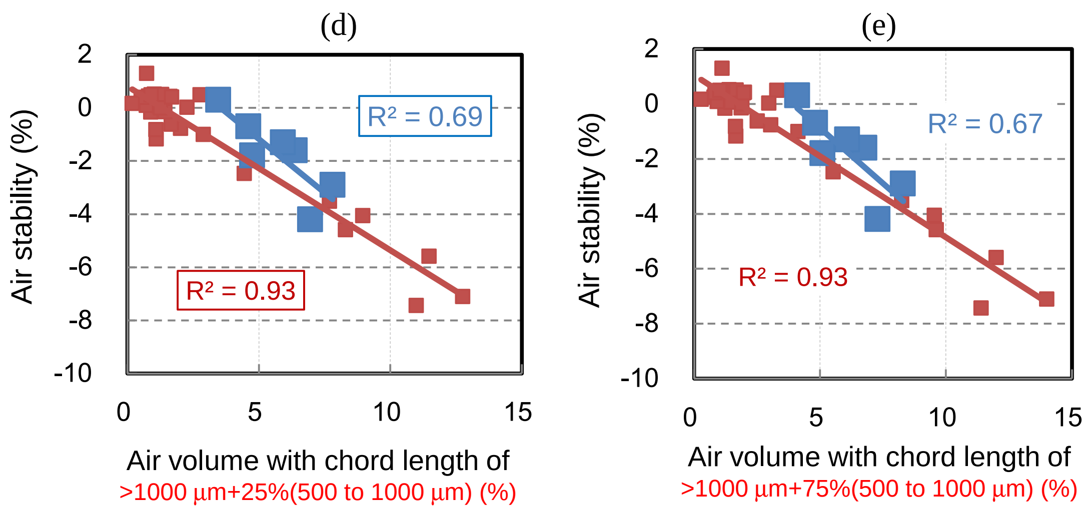



**Fig.2.15 Relationship between the stability of air and the air volume in concrete and mortar at fresh stage with chord length >1000 µm, >500 µm or >1000 µm+25%, 50% or 75% of (500 to 1000 µm)**

These 7 points of concrete test result was plotted close to the correlation in mortar tests as shown in the figure. Interestingly, the concrete test results well fit with the correlation of mortar tests which indicated that the critical size and the consideration on the unification of air bubbles in mortar experiment can also be well applied to the concrete experiment.

## 2.5. Summary

Instability in volume of air in fresh mortar caused by the existence of air bubbles with chord length of mainly over 1000 µm and partially 500 µm to 1000 µm due to unification of air bubbles. This summary is shown in **Fig. 2.16** as follow. To ensure the stability of air and to reduce the floating bubbles that may lead to a risk of escaping, the critical size of 500 µm was set up for discussion from **CHAPTER 3**.

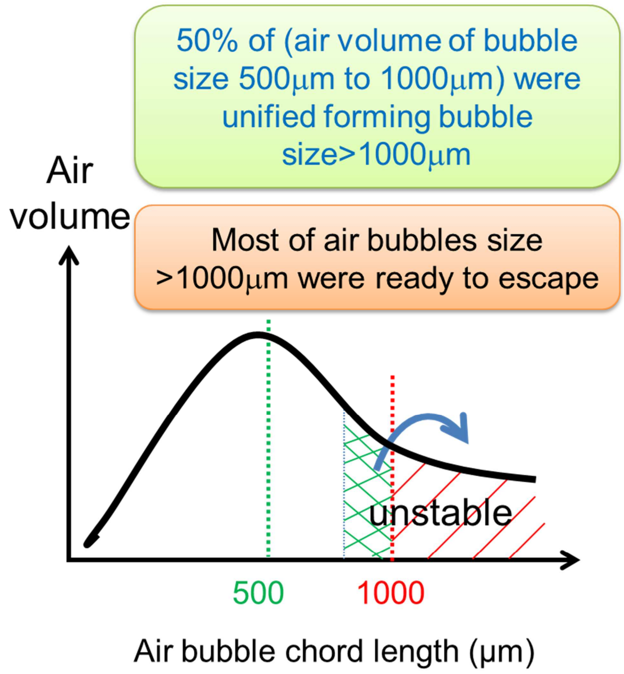



**Fig.2.16 Summary of the border of air bubbles size causing instability of air volume**

# CHAPTER 3 EFFICIENCY IN ENTRAINING VOLUME OF AIR DEPENDING UPON VISCOSITY SUBJECTED TO TYPE OF MIXING PROCEDURE

## 3.1. Purpose

The purpose of this chapter is to show that viscosity subjected to different types of mixing procedure cause the difference in the formation of air volume of coarse or fine size of air bubbles. This clarification can enable modifying the mixing procedure in the right direction thus enable minimizing the air volume of coarser bubbles and improving the stability of air.

## 3.2. Hypothesis

The viscosity of the mixture during mixing procedure for entraining air bubbles could be a determinant factor deciding the fineness of air distribution. Even though higher viscosity of mixture could reduce the rate of escaping air bubbles, the stability of air would not able to be improved if the air volume of coarse size bubbles was high. Since the target of this study is to ensure the stability of air, the finer the air system is preferable. It was better to initially entrain finer air entrainment to enhance the stability of air rather than to allow excessive existence of coarse air volume which was expected to come with the high viscosity mixture. The assumption on the effect of varied viscosity on the capacity in entraining coarse or fine air bubbles is shown in **Fig. 3.1**.

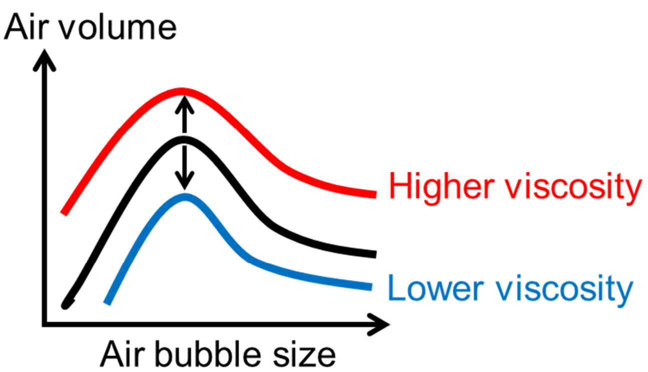



**Fig.3.1 Viscosity influenced the capacity of entraining air volume**

## 3.3. Difference in Viscosity between Dividing Water Mixing Procedure and Simple Mixing Procedure

### 3.3.1. Experimental plan to clarify the affected of dividing water on viscosity

In mixing procedure B, the mixing water was divided into two portions. In this section, the portion of water firstly poured with SP was varied to clarify that the dividing water was contribute to the effectiveness of mixing procedure and that there existed the optimum portion of water to be poured with SP to lower viscosity of the mixture. The viscosity of the mixture (evaluated through the term funnel speed in this study), was used to indicate the effectiveness of divided portion of mixing water. It was assumed that the highest the funnel speed was able to produce the finest air distribution. As it was observed in the **Chapter 1**, the mixing procedure B (the water-dividing mixing procedure) was usually accompanied with the higher funnel speed of mortar and as the result produced the finer air distribution than mixing procedure A (the simple mixing procedure).

In this section, the target of experiment is to prove that the portion of water firstly poured with SP and mixed was essential to change the characteristics of the mixture before pouring AE. The dispersing action of SP lower the viscosity of the mixture resulting in higher funnel speed could entrain finer air bubbles system. Mortar experiments were conducted with mixing procedure A and B with and without AE. In case of the mixing procedure B, different portion of water pouring with SP was varied by $W_1$* (the total water content poured before adding AE) at 40%, 35%, 30%, 25%, 20%, or 15% of cement weight. Mixing procedures conducted in this section is shown in **Figure 3.2**.

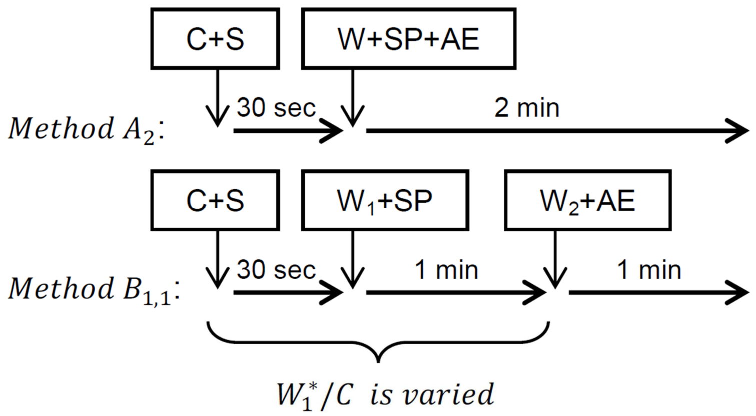



**Fig.3.2 Different types of mixing procedure to clarify viscosity due to water dividing mixing**

The materials used for experiment in this chapter is shown in **Table 3.1**

**Table 3.1 Materials used for mortar experiment in Chapter 3**

| | | |
|---|---|---|
| Cement (C) | | Ordinary Portland cement (3.15g/cm$^3$) |
| Fine aggregate (S) | | Crushed limestone sand (2.68g/cm$^3$, F. M. 2.7) |
| Superplasticizer (SP) | $SP_1$ | Polycarboxylic based with viscosity agent |
| | $SP_4$ | Polycarboxylic based |
| Air entraining agent (AE) | $AE_1$ | Alkyl ether-based anionic surfactants *(AE: water concentration is 1:99)* |

### 3.3.2. Portion of dividing water affected viscosity of mixture

The relationship between the portion of water firstly poured and mixed with SP and the funnel speed of mortar obtained after mixing in case of mixing procedure A or B, with and without AE, is shown in **Fig. 3.3**.

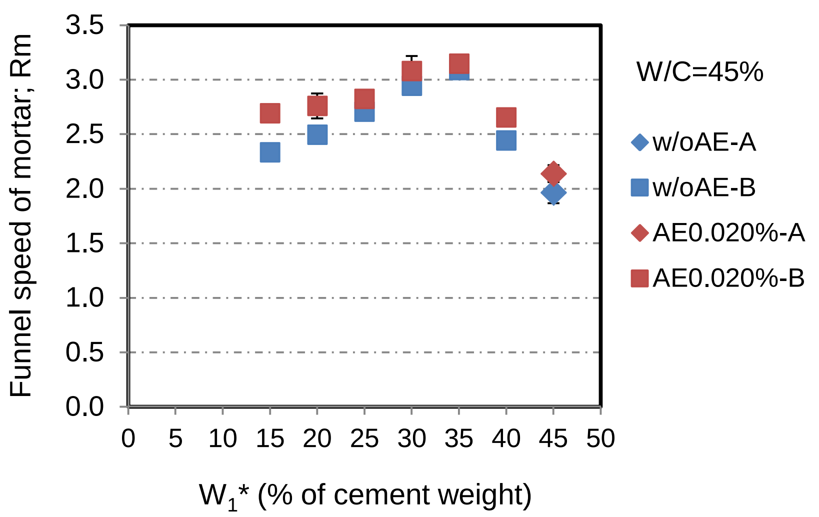



**Fig.3.3 Dividing water on funnel speed of mortar produced with or without AE**

It can be seen that with the first portion of water in between 30% and 35% of cement weight ($W_1$*=$W_1$+SP+surface water of the fine aggregate), the funnel speed reached their highest value and was largely faster than that of mortar produced with mixing procedure A. Furthermore, when dosage of AE of 0.020% of cement weight was used, the funnel speed was slightly increased in spite of the increased in air volume. This result clearly explained that the effect of dividing water was significantly affected the viscosity of the mixture. Thus the lower viscosity of mixture obtained with mixing procedure B could be a dominant factor entraining finer air entrainment by limiting the formation of air volume of coarser bubbles.

## 3.4. Varied Viscosity of Mixture Generated Different Water Content

### 3.4.1. Experimental plan to re-appear the characteristic of mixing procedure A to a lower water content mixture of mixing procedure B

In this section, air size distribution of mortar conducted with different water content (W/C of 35%, 40% and 45% of cement) with mixing procedure B was analyzed and was compared to that produced by mixing procedure A at W/C of 40% and 45% of cement weight. The mixing time with AE for both mixing procedures in this section was chosen to be 2 minutes (Y=2min). The mixing procedure of these methods is shown in **Fig. 3.4**. The mix-proportion and characteristic of mortar at fresh stage is shown in **Table 3.2**.

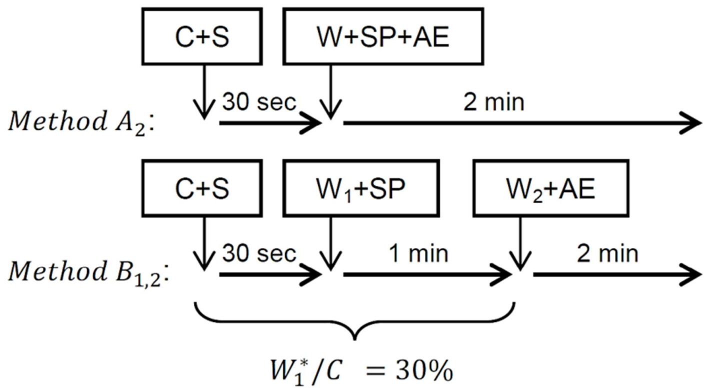



**Fig.3.4 Mixing procedure $A_2$ and $B_{1,2}$**

**Table 3.2 Mix-proportion and properties of mortars produced with mixing procedure A and B at different water content**

| W/C(% by weight) | Mixing procedure | $SP_1/C$(%) | $AE_1/C$ (%) | Flow (mm) | Funnel speed | Initial air content (%) | Air content in 2h (%) |
|---|---|---|---|---|---|---|---|
| 35 | $B_{1,2}$ | 2.0 | 0.010 | 245 | 1.38 | 22.4 | 17.1 |
| 40 | $B_{1,2}$ | 1.4 | | 247 | 2.44 | 13.0 | 12.9 |
| 45 | $B_{1,2}$ | 1.1 | | 236 | 3.32 | 6.4 | 6.9 |
| 40 | $A_2$ | 2.0 | | 253 | 1.98 | 14.9 | 13.0 |
| 45 | $A_2$ | 1.4 | | 252 | 2.65 | 13.0 | 10.5 |

### 3.4.2. Viscosity generated with different types of mixing procedure affecting the air distribution

With mixing procedure B, lower content of water required higher dosage of AE to reach the similar mortar flow. The lower water content mixture having higher viscosity (indicated by the funnel speed value) entrained higher content of air. The stability of air was gradually decreased when the water content was reduced which would be caused by the increase in the air volume of coarse size bubbles. Mortar produced with mixing procedure B at W/C 40% acted similarly as mixing procedure A at W/C 45% as shown in **Fig. 3.5**. They required the same dosage of SP to reach the similar mortar flow then possessed similar funnel speed and produced the same volume of air.

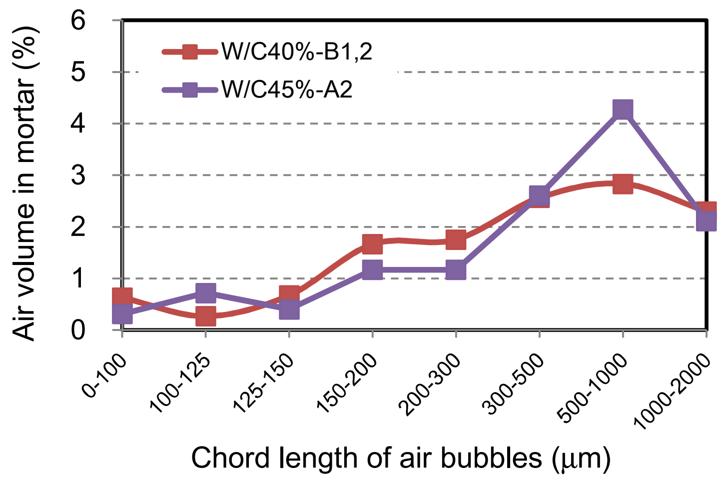



**Fig.3.5 Air size distribution in mortar produced with mixing procedure B at W/C 40% and with mixing procedure A at W/C 45%**

With mixing procedure B, higher viscosity mixture entrained higher volume of air at all size of bubbles. Air size distribution of mortar produced with mixing procedure A at W/C 45% was similar to that produced with mixing procedure B at W/C 40%. This result explained that the difference in viscosity between mixtures determined how air bubbles would be entrained. The funnel speed of mortar produced with mixing procedures A and B at different water content is shown in **Fig. 3.6**.

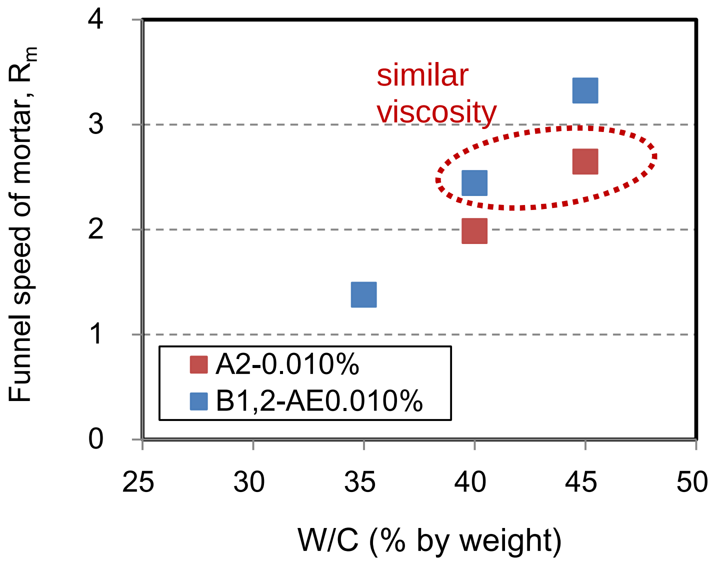



**Fig.3.6 Viscosity of mortar due to different water content and mixing procedure**

Lower water content resulted in higher viscosity and mixing procedure B usually produced higher funnel speed than that with the mixing procedure A. Following hypothesis explaining above, it was expected that with similar viscosity, the capacity of air entrainment would be similar. To prove this hypothesis, the relationship between funnel speed of mortar and the air volume of coarse air (defined as air bubbles with chord length of larger than 500μm) or the air volume of fine air (defined as air bubbles with chord length of smaller than 500μm) are shown in **Fig. 3.7** and **Fig. 3.8** respectively.

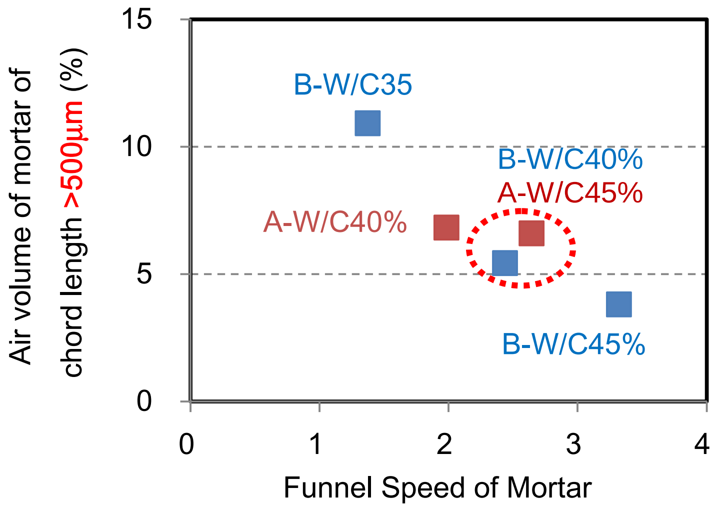



**Fig.3.7 Relationship between viscosity from different types of mixing procedure with different water to cement ratio on entraining coarse air volume**

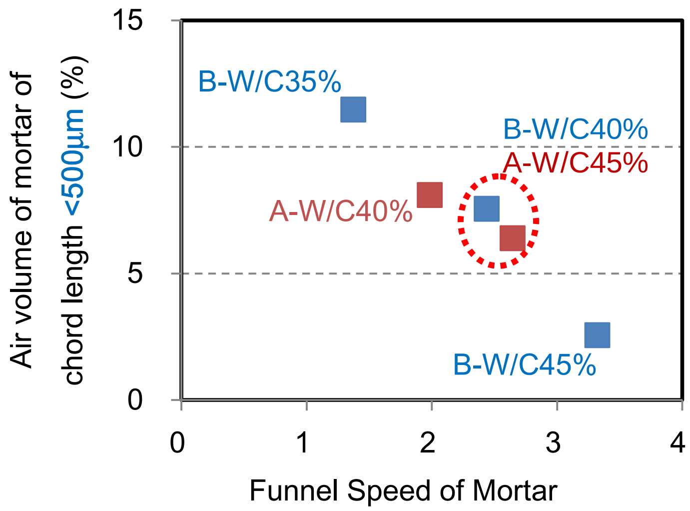



**Fig.3.8 Relationship between viscosity from different types of mixing procedure with different water to cement ratio on entraining fine air volume**

It can be found that with either mixing procedure, higher viscosity had higher capacity in entraining air volume of either coarse or fine bubbles. In spite of the different types of mixing procedure, the index for (funnel speed of mortar) viscosity indicated well the difference in air volume since all the plots formed a good correlation. For example, as the mixture produced with mixing procedure A at W/C of 45% and with mixing procedure B at W/C of 40% had similar viscosity, the two mixtures possessed the similar air volume of either coarse or fine bubbles. This result sincerely explained that the viscosity was the key factor for differentiating the air entrainment characteristics.

Lowering the viscosity of mixture during mixing was beneficial to reduce the air volume of coarse size bubbles, thus the mixing procedure B was effective. More importantly, with mixing procedure B, there was more room for improvement in the fineness of air entrainment through increasing dosage of AE. Higher viscosity mixture entrained higher volume of air which means the dosage of AE required was less than the lower viscosity mixture to reach the similar air volume. This result proved that the difference in entraining air size distribution between the mixing procedure A and B was mainly due to the difference in viscosity which was a significant factor determine the entrainment of coarse or fine size air volume. The effectiveness of higher AE dosage is discussed in the following chapter.

## 3.5. SUMMARY

From the results and data analysis mentioned above, summary of this chapter can be written as follows:

1) Viscosity (in terms of the funnel speed of mortar) from different types of mixing procedure was considerably different. An effective mixing procedure (water dividing mixing procedure) produced a lower viscosity mixture than that produced with a simple mixing procedure. The difference in viscosity between these types of mixing procedure determined its effectiveness in entraining fineness of air and its capacity in entraining the total air volume.

2) Lower viscosity of mixture entrained lower air volume but at the same time eliminating the volume of coarse size air bubbles which is beneficial in improving the stability of air. With an effective mixing method, fineness of air entrainment could be more improved through higher dosage of AE. The efficiency in volume of air entrainment subject to viscosity is shown in **Fig. 3.9**.

3) Water-dividing mixing procedure can be re-appeared by simple mixing procedure at lower water to cement ratio.

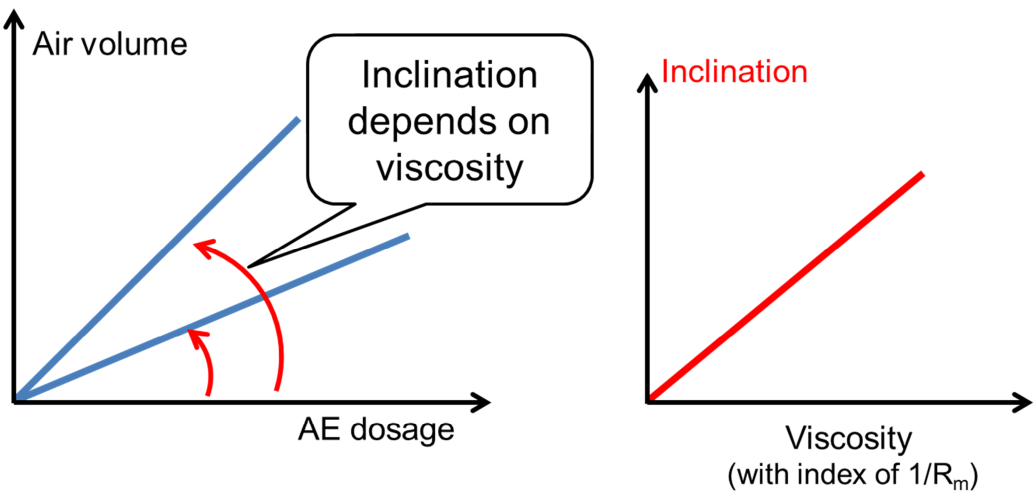



**Fig.3.9 Definition of index for efficiency in volume of air entrainment subject to viscosity**

# CHAPTER 4 REPLACEMENT OF ENTRAINING FINE AIR BUBBLES WITH COARSE ONES DEPENDING UPON VISCOSITY AND DOSAGE OF AIR-ENTRAINING AGENT

## 4.1. Purpose

The purpose of this chapter is to clarify the effect of dosage of AE on entraining coarse or fine size air bubbles. In addition, the combination effect of the viscosity of the mixture subject to different types of mixing procedure and the dosage of AE on the upper limit volume of fine air is to be clarified.

## 4.2. Hypothesis

It is known that AE agent is used to stabilize air entrainment system [13]. With sufficient dosage of AE, finer air bubbles would be able to stay in the system for longer time. On the other hand, with shortage of AE, those fine air bubbles entrained during mixing would unify and form a larger size bubbles and then collapse. This phenomenon would cause the increase in coarse size air volume provoking the damage to the stability of air. The expectation of on the influence of dosage of AE agent on the volume of coarse air bubbles is shown in **Fig. 4.1**.

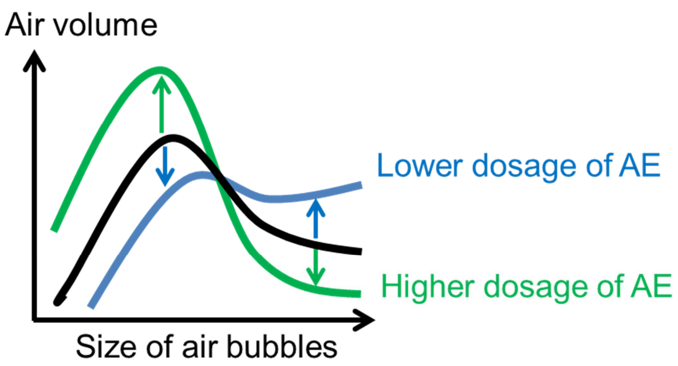



**Fig.4.1 Influence of dosage of AE agent on the volume of coarse and fine bubbles**

Viscosity of the mixture could be a significant factor defining the required dosage of AE to reach the target volume of air. Even with the same total air volume of two mixtures with different viscosity, the air entrainment could be different. As shown in the previous chapter(s), higher viscosity of the mixture produced higher air volume than that of the lower viscosity. Thus, it was obvious that the lower viscosity mixture would require higher dosage of AE to entrain similar air volume as that of the higher viscosity. Requirement of higher dosage of AE could be beneficial in entraining higher volume of fine air and minimizing the volume of coarse air. Thus, the combination of viscosity and dosage of AE could determine the air volume of coarse and fine bubbles to be entrained.

## 4.3. Sufficient or Shortage of Dosage of Air Entraining Agent on Minimizing Coarse Size Air Bubbles

Before discussing the upper limit volume of fine air, this section simply discussed on the effect of dosage of AE and mixing time on entraining coarse air bubbles in the cases of mixing procedure A and B respectively.

### 4.3.1. Effect of air entraining agent dosage on air size distribution of mortar

Mortar samples tested in this chapter were produced at different dosage of AE with different types of mixing procedure including $A_Y$ and $B_{1,Y}$ as shown in **Fig. 4.2**.

In case of the mixing procedure $A_2$, three mortar samples at dosage of AE of 0.005%, 0.010% and 0.020% of cement weight were chosen. In case of mixing procedure $B_{1,4}$, three mortar samples at dosage of AE of 0.010%, 0.020%, and 0.040% were chosen. The mix-proportion and the properties at fresh stage of these samples are shown in **Table 2.1** and **2.2**.

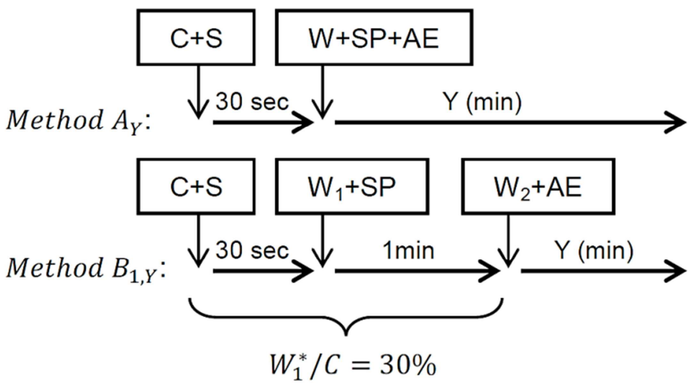



**Fig.4.2 Different types of mixing procedure to clarify the effect of mixing time with AE (Y) at different dosage of AE**

The effect of dosage of AE on air size distribution of mortar samples produced with method $A_2$ is shown in **Fig. 4.3**.

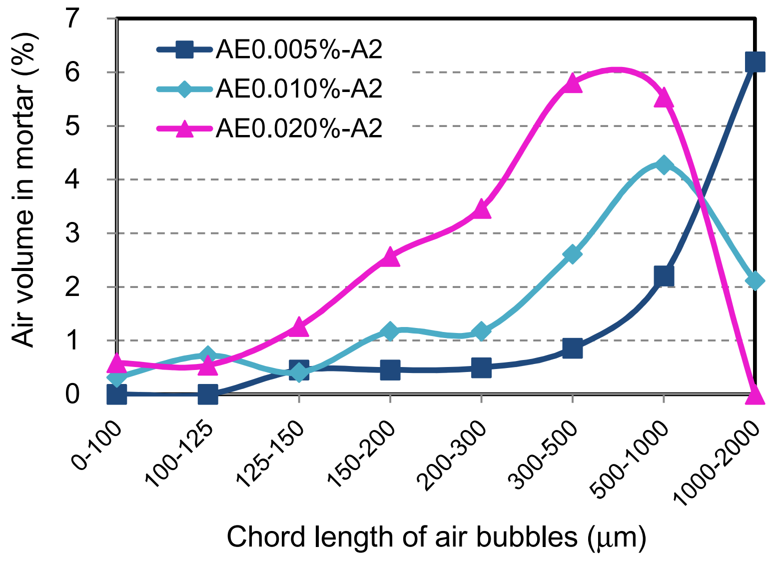



**Fig.4.3 Air size distribution of mortar with increasing dosage of AE produced with mixing procedure $A_2$**

Higher dosage of AE significantly increased the air volume of bubble with chord length of smaller than 1000µm while decreasing the air volume of bubble with chord length of larger than 1000µm. This result showed a beneficial influence of dosage of AE on the fineness of air bubbles. Shortage of dosage of AE entrained large content of coarse size air bubbles. This result can be explained that during mixing, fine size air bubbles were unified and formed a larger one when the dosage of AE was not sufficient to stabilize those at that fine size. As a result, coarse size air content increased and escaped.

In case of mixing procedure B (lower viscosity), higher dosage of AE was required to reach the similar air volume as mixing procedure A. With the mixing time with AE of 4 minutes as shown in **Fig. 4.4**, increase in AE dosage increased significantly the content of fine size air bubbles while decreasing the air volume of coarse size air bubbles.

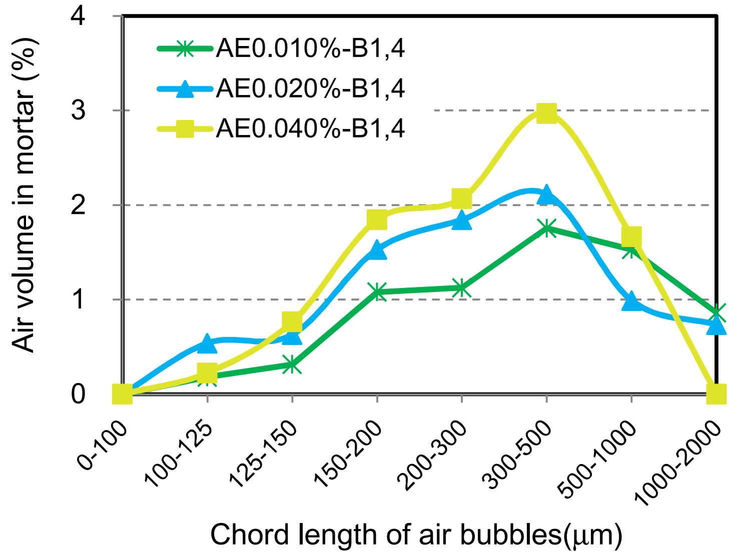



**Fig.4.4 Air size distribution of mortar with increasing dosage of AE produced with mixing procedure $B_{1,4}$**

### 4.3.2. Influence of longer mixing time on air size distribution of mortars

Three mortar samples produced with mixing procedure $A_Y$ with Y of 2 minutes, 4 minutes or 6 minutes with the same mix-proportion of AE dosage of 0.010% of cement weight were compared. The effect of longer mixing time with mixing procedure A on air size distribution of mortar samples is shown in **Fig. 4.5**.

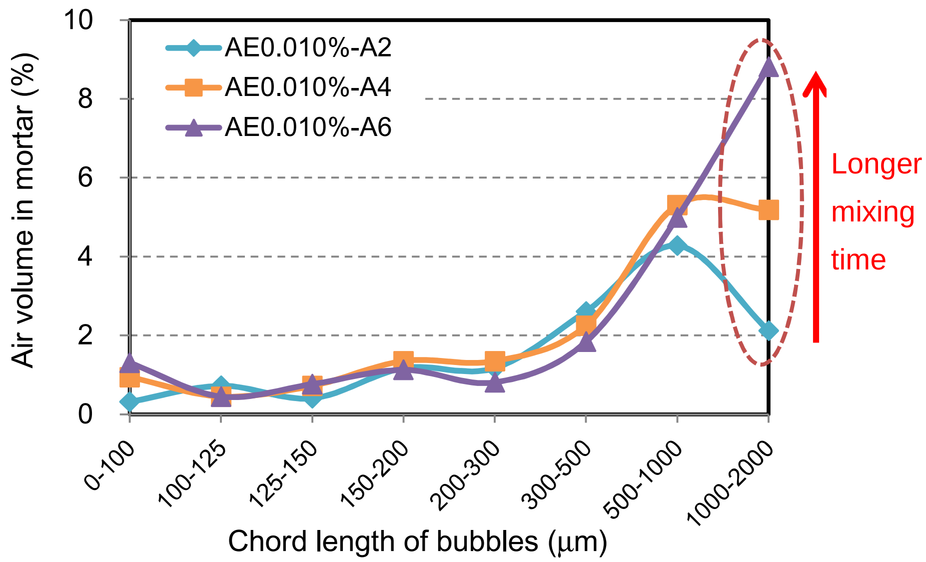



**Fig.4.5 Effect of mixing time on air size distribution produced with mixing procedure $A_Y$ at dosage of AE of 0.010% of cement weight**

In this case, with longer mixing time, air volume was significantly increased of bubbles with chord length of larger than 1000 μm. On the other hand, the air volume of bubble with chord length of smaller than that was not considerably changed with the prolonging of mixing time. This result explained that longer mixing time with mixing method A did not improve the fineness of fine air bubbles and instead caused an increase in coarse air volume. This case can also be due to the shortage of AE dosage which caused the air entrainment to reach the upper limit of fine air volume at shorter mixing time of AE.

Air size distribution of three mortar samples produced at the same mix-proportion of AE dosage of 0.010% of cement weight with mixing procedure $B_{1,Y}$ in which Y was varied by 2 minutes, 4 minutes or 6 minutes is shown in **Fig. 4.6**.

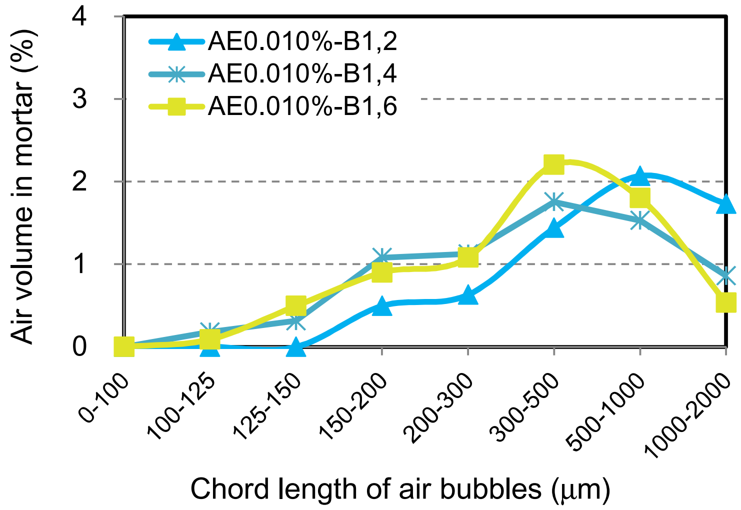



**Fig.4.6 Effect of mixing time on air distribution produced with mixing procedure $B_{1,Y}$ at dosage of AE of 0.010% of cement weight**

On the other hand, longer mixing time with AE with mixing procedure B increased the air volume of fine air bubble while slightly decreasing that of coarse air bubbles. With lower viscosity mixture, increasing mixing time with AE did not harmfully increase the coarse air bubbles.

Air size distribution of four mortar samples produced at the same mix-proportion of AE dosage of 0.020% of cement weight with mixing procedure $B_{1,Y}$ in which Y was varied by 2 minutes, 4 minutes, 6 minutes, or 10 minutes is shown in **Fig. 4.7**.

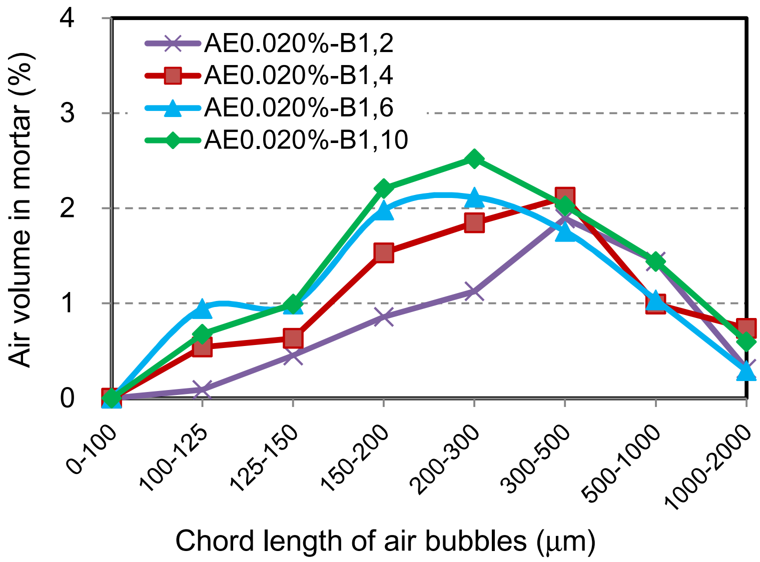



**Fig.4.7 Effect of mixing time on air size distribution produced with mixing procedure $B_{1,Y}$ at dosage of AE of 0.020% of cement weight**

Similarly to that of lower dosage of AE case, longer mixing time with AE increased the air volume of fine bubbles while the air volume of coarser bubbles was not disturbed. This result showed the efficiency of mixing procedure B in adjusting for target air volume by prolonging the mixing time without causing any unfavorable effect to the stability of air. Furthermore, longer mixing time with AE was beneficial in case of method B in order to improve the fineness of air distribution without any harmful effect to the stability of air

### 4.4. Hypothesis on Upper Limit Volume of Fine Air

Through the test results mentioned above, hypothesis on upper limit volume of fine air capable to be entrained with longer mixing time was set up as shown in **Fig. 4.8**. With increase in mixing time with AE, there are three phases of air entrainment occurred.

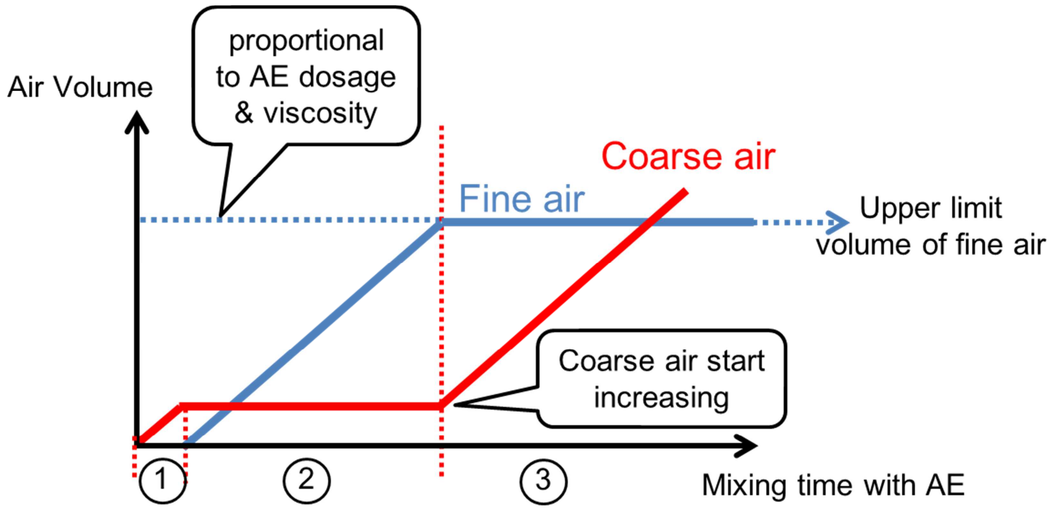



**Fig.4.8 Upper limit volume of fine air proportional to dosage of AE and viscosity with longer mixing time**

In phase**"1"**, coarse air volume is entrained to some extent.

In phase **"2"**, coarse air volume is posed to be increased while fine air volume would start increasing. In this phase, the entrapped coarse bubbles are actually replaced with finer bubbles by shearing action.

In phase **"3"**of mixing, fine air reaches the upper limit volume and coarse air bubbles start increasing again. This phase starts when the dosage of AE has been expired and then unifications between air bubbles started occurring faster than the shearing air bubbles causing an increase in coarse air volume and limiting the volume of fine air to the upper limit volume.

In phase **"3"**,it is more likely that if the mixing time is excessive, the fine air volume could be possible to be decreased after reaching the upper limit volume and some of the coarse air bubbles could be already escaped before finishing mixing resulted in a lower total air volume.

At low dosage of AE, the upper limit of fine air could be reached with shorter mixing time and the coarse air volume could be high. In this case, when the coarse air volume starts increasing, the upper limit volume of fine air is reached. On the other hand, at higher dosage of AE and if mixing time is not excessive, the coarse air volume could be minimized.

## 4.5. Upper Limit Volume of Fine Air depending upon Dosage of Air-Entraining Agent and Viscosity

### 4.5.1. Upper limit volume of fine air by different types of mixing procedure

The volume of entrained coarse air volume (of bubbles with chord length of >500 µm) and fine air volume (of bubbles with chord length of <500 µm) of mortar samples produced at different dosage of AE are shown in **Fig. 4.9** and **Fig. 4.10** in case of mixing procedure A and B respectively.

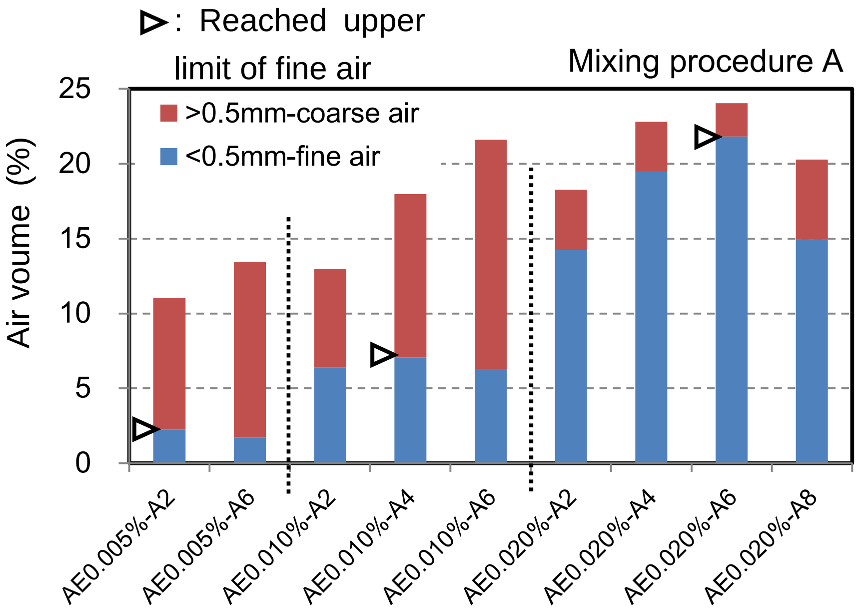



**Fig.4.9 Volume of entrained coarse and fine air respectively at different dosage of AE in case of mixing procedure A**

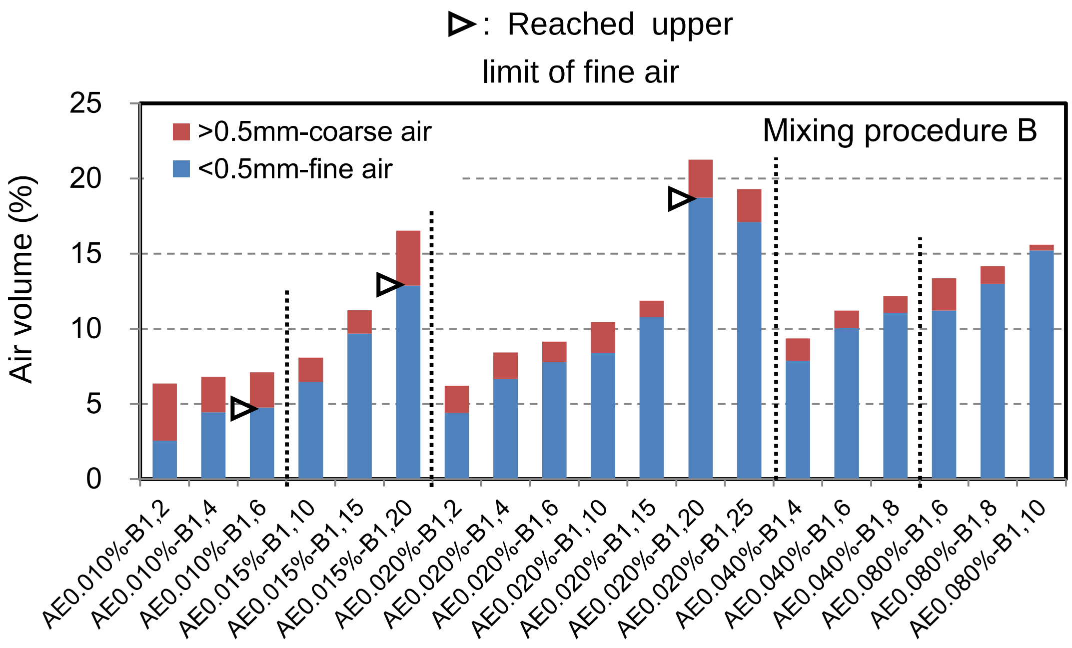



**Fig.4.10 Volume of entrained coarse and fine air respectively at different dosage of AE in case of mixing procedure B**

In the case of mixing procedure A, at dosage of AE of 0.005% or 0.010% of cement weight, air volume of fine bubble was not able to be increased with longer mixing time, in which the upper limit volume of fine air was reached. When the upper limit of fine air was reached, increasing mixing time increased the air volume of coarse bubbles instead. When the dosage of AE was 0.020%, air volume of fine bubbles started to be increased with longer mixing time while the air volume of coarse bubbles kept its similar volume with the increase in mixing time. This can be explained that, with enough dosage of AE, fine air bubbles were again available to be formed while the rate of unification between air bubbles was restrained to some limit volume.

In the case of mixing procedure B, from the dosage of AE of 0.010% of cement weight, the air volume of coarse bubbles was not increased with longer mixing time with AE while the air volume of fine bubbles was continually increased. With lower viscosity, mixing procedure B could effectively restrain the formation coarse air bubbles and the upper limit volume of fine air bubbles would be reached at longer mixing time than the mixing procedure A. This result explained that lower viscosity of mixture was beneficial to hinder the entrainment of coarse air bubbles.

At lower dosage of AE, the air volume of coarse bubbles was higher and the coarse air bubbles were able to be minimized by increasing the dosage of AE. This phenomenon was more obvious in case of the mixing procedure A. At each dosage of AE, longer mixing time with AE could entrain higher air volume of fine bubble to some limit depending on the viscosity of the mixture and the dosage of AE. In case of mixing procedure A, which normally produced higher viscosity mixture, the total air volume was higher than that of the mixing procedure B. Taking the case of AE dosage of 0.020% of cement weight as an example, with the same mixing time of 6 minutes, the total air volume was 24.0% and 9.2% in case of the mixing procedures A and B respectively.

On the other hand, when the mixing time was kept increasing, the fine air volume in case of mixing procedure B could reach the upper limit volume at 18.7% with the mixing time of 20 minutes. If the mixing time was increased to 25 minutes, both the total air volume and the fine air volume were decreased. This phenomenon explained that with an excessive duration of mixing time, the unification between air bubbles was faster than the shearing of bubbles forming finer air. Thus, this was resulted in decrease of the fine air volume and possibly also of the total air volume since the coarse air bubbles were ready to escape before finishing mixing as the dosage of AE was no longer effective to stabilize those air bubbles.

This phenomenon can also be observed in case of the mixing procedure A at the dosage of AE of 0.020% of cement weight. With the mixing time of 8 minutes, both the fine air volume and the total air volume were decreased indicating that the upper limit volume of fine air was ready reached at the mixing time of only 6 minutes. It was interesting to note that, the upper limit volume of fine air reached at considerably shorter mixing time in case of the mixing procedure A than mixing procedure B due to the difference in viscosity between these two mixing procedures.

### 4.5.2. Correlation of upper limit of fine air and the combination of viscosity and dosage of air-entraining agent

The relationship between dosage of AE and the air volume of fine bubbles with longer mixing time is shown in **Fig. 4.11**.

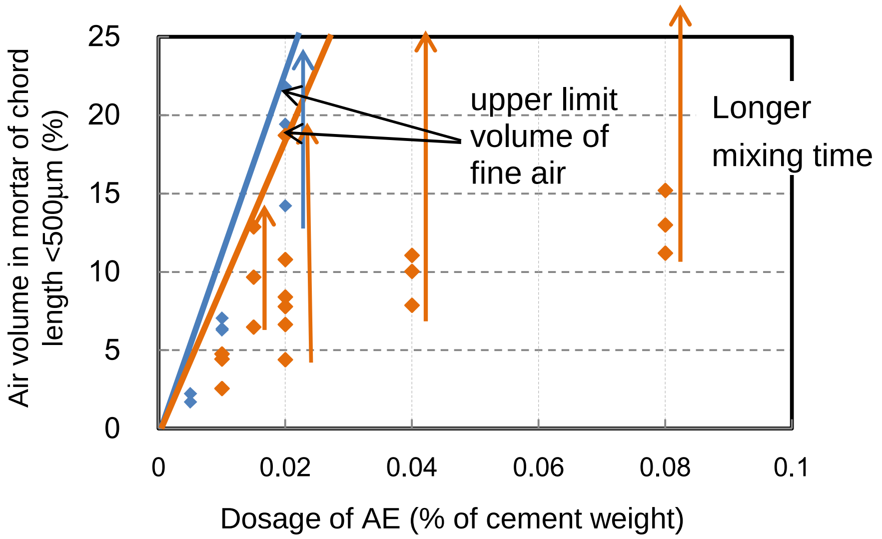



**Fig.4.11 Upper limit volume of fine air increased with AE dosage and depended on viscosity of mixture**

The capacity in entraining air volume was varied with viscosity subject to different mixing procedure and the dosage of AE. The upper limit of fine air volume was reached at shorter mixing time in case of mixing procedure A than in case of mixing procedure B. While the upper limit of fine air was reached, the coarse air volume started increasing when mixing time was increased as shown in **Fig. 4.12**.

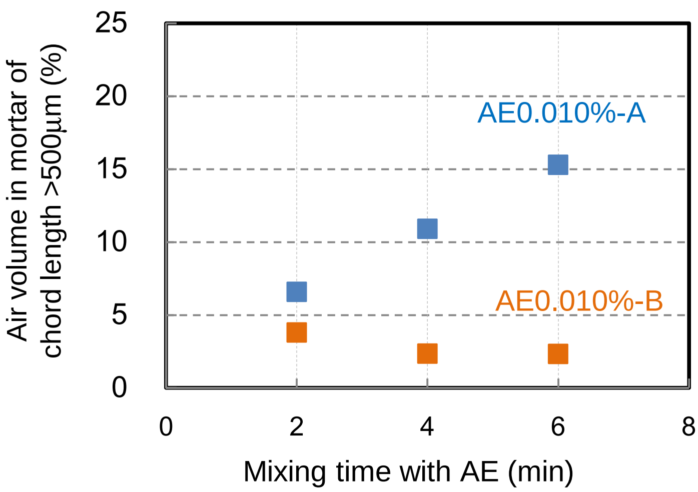



**Fig.4.12 Volume of coarse air increased when the upper limit volume of fine air was reached with mixing procedure A**

This was an example of a low dosage of AE of 0.010% of cement weight. It can be seen that, the coarse air volume kept increasing with longer mixing time in case of mixing procedure A as the fine air volume was not able to increased anymore. On the other hand, the change in coarse air volume was small in case of the mixing procedure B as the fine air volume can be increased to some more extend.

Also, the linear correlation between the combination of viscosity and dosage of AE and the upper limit of fine air volume was obtained as shown in **Fig. 4.13**.

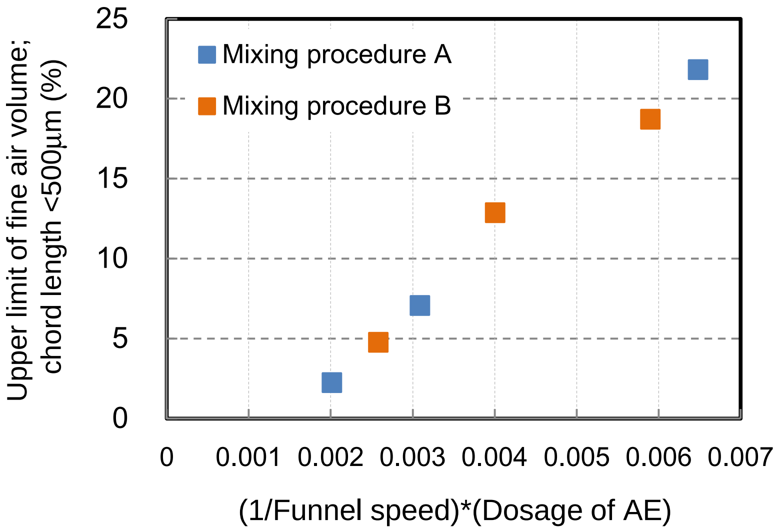



**Fig. 4.13 Unique relationship between viscosity & dosage of AE and the upper limit volume of fine air**

This result indicated that the upper limit volume of entrained fine air was closely related to both the viscosity of mixture (which differentiate considerably by different mixing procedure) and the dosage of AE.

## 4.6. Summary

1) Sufficient dosage of AE could minimize the air volume of coarse size air bubbles while increasing the air volume of fine size bubbles. Shortage of AE resulted in the faster unification of fine size air bubbles thus increasing the air volume of coarse size air bubbles.
2) With an effective mixing method, sufficient dosage of AE and increasing mixing time with AE increased the air volume of finer size air bubbles while causing no

increase in air volume with coarser size.

3) The upper limit volume of entraining fine air depends on the dosage of AE and the viscosity of the mixture produced with different types of mixing procedure. As long as the upper limit volume of fine air was not reached, longer mixing time generated more air volume of fine bubbles without increasing that of the coarser size. In contrast, after the upper limit volume of entraining fine air was reached, increasing mixing time with AE caused the increase in air volume of coarse bubbles which was harmful to the stability of air.

# CHAPTER 5 CONCLUSION AND FURTHER PERSPECTIVE RESEARCH

The purpose of this study is to modify the mixing procedure to effectively improve the stability of air bubbles and to reach target air volume with satisfied fineness of air entrainment. To improve the stability of air, the air volume of coarse bubbles has to be reduced. With measurement the air size distribution with AVA of fresh mortar samples with various mix-proportion and different types of mixing procedure for determining the critical size of air which escapes either by collapsing or by floating. The influence of viscosity and dosage of AE on entraining fine air volumes were also clarified. Finally, the replacement of entraining fine air bubbles with coarse ones was possible to be conducted.

Conclusion drawing form this study can be written as following:

1) Instability in volume of air in fresh mortar of self-compacting concrete caused by the existence air bubbles with chord length of mainly over 1000μm and partially 500 μm to 1000μm due to unification between air bubbles.
2) Higher viscosity of fresh mortar during air entrainment in mixing resulted in higher efficiency in volume of air entrainment due to the difference in mixing procedure. It reappeared with difference in water to cement ratio with the similar mixing procedure.
3) The upper limit volume of air entrainment of fine air with chord length of less than 500μm was found to be in proportional to the dosage of air-entraining agent. After reaching the upper limit volume of fine air as mixing time passed, the volume of coarse air started increasing.

For a further study, various properties of materials should be conducted to promote the reliability on the value of critical size of air bubbles to determine.

# LIST OF FIGURES

# LIST OF TABLES

# REFERENCES

[1] "Recommendation for self-compacting concrete, "*JSCE Concrete Engineering*, Series 31,1999.

[2] Ozawa, K. et al., "Development of high performance concrete based on the durability design of concrete structures," *Proceedings of the second East Asia and Pacific Conference on Structural Engineering and Construction (EASEC-2),* 1989, 1, pp 445-450.

[3] Okamura, H., Maekawa, K., and Ozawa, K., "High Performance Concrete," *Gihodo Publishing*, 1993.

[4] Maekawa, K. and Ozawa, K., "Development of SCC's prototype," (in Japanese), Self-Compacting High-Performance Concrete, *Social System Institute,* pp 20-32.

[5] Okamura, H. and Ozawa, K., "Mix-design for self-compacting concrete," *Concrete Library of JSCE*, 25, pp 107-120.

[6] Okamura, H. and Ouchi, M., "Self-compacting concrete," *Journal of advanced concrete technology*, 2003 1(1)pp5-15.

[7] Lianxiang, Du. and Kevin, J. F., "Mechanism of air entrainment in concrete, "*Cement and Concrete Research, ELSEVIER*, 2005, 35, pp 1463-1471.

[8] Chatterji, S. and Gudmundsson, H., "Characterization of entrained air bubble systems in concretes by means on an image analyzing microscope," *Cement and Concrete Research,* 1977, 7, pp 423-428.

[9] ACI C 318, "Building code requirements for structural concrete and commentary," *ACI Committee 318 Report, ACI,* 2002.

[10] Steven, H. K. and Michelle, L. W., "Design and control of concrete mixtures, "*PCA 15th edition*, 2011.

[11] Neville, A. M., "Properties of concrete," 4th edition, 1995.

[12] Power, T. C., "Topics in concrete technology – Part 3: Mixtures containing intentionally entrained air," *J. PCA Res. Dev. Labs*, 1964, 6(13), pp 19-42.

[13] Mielenz, R. C., "Use of surface active agents in concrete," *Proceeding of the Fifth International Symposium of Chemistry of Cement,* Principal paper session IV-1, 1968, pp. 1-29.

[14] Gebler, S. and Klieger, P. "Effect of fly ash on the air-void stability of concrete," *ACI Special Publication*, 1983, SP-79, pp 103-120.

[15] Gay, F. T., "The influence of mixing temperature on air content of hardened concrete,"

*Proceeding of the Fifth International Conference of Cement Microscopy*, 1983, pp. 231-239.

[16] Gay, F. T., “The influence of thermal history on air content and air void system parameters of concrete of differing cement factors,” *Proceeding of the Sixth International Conference of Cement Microscopy*, 1985, pp. 384-401.

[17] Pistilli, M. F., “Air-void parameters developed by air entraining admixtures as influenced by soluble alkalies from fly ash and Portland cement,” *ACI Materials Journal*, 1983, 80(3), pp 217-222.

[18] Khayat, K. H., “Optimization and performance of the air-entrained, self-consolidating concrete,” *ACI Materials Journal*, 2000, 97(5), pp 526-535.

[19] Khayat, K. H. and Assaad, J., “Air-void stability in self-consolidating concrete,” *ACI Materials Journal*, 2002, 99(4), pp 408-416.

[20] Kobayashi, M. et al., “Frost resistance of superplasticized concrete,” *ACI special Publication*, 1981, SP-68, pp 269-282.

[21] Szwabowski, J. and Łaźniewska-Piekarczyk, B., “Air-entrainment problem in self-compacting concrete,” *Journal of Civil Engineering and Management*, 2009, 15:2, pp 137-147.

[22] Fagerlund, G., “Air-pore instability and its effect on the concrete properties,” *Nordic Concrete Research*, 1990, 9, pp 39-52.

[23] “ASTM dictionary of engineering science and technology,” 2005, 10$^{th}$ edition, sponsored by ASTM Committee E02 on Terminology.

[24] “Standard Specifications for Concrete Structures,” *JSCE Guidelines for Concrete*, 2007.

[25] MacInnis, C. and Racic,D,“The effect of superplasticizers on the entrained air-void system in concrete, ”*Cement and Concrete Research*, 1986, 16, pp 345-352.

[26] Khatib, J. M. and Mangat, P. S., “Influence of superplasticizer and curing on porosity and pore structure of cement paste,” *Cement and Concrete Composites*, 1999, 21, pp 431-437.

[27] Saucier, F., Pigeon, M., and Cameron, G., “Air-void stability, Part V: temperature, general analysis and performance index,” *ACI Materials Journal*, 1991, 88(1), pp 25-36.

[28] Daimon, M. and Roy, D. M., “Rheological properties of cement mixes: I. Methods, preliminary experiments, and adsorption studies,” Cement and Concrete Research, 1978, 8(6), pp 753-764.

[29] Thomas, M. D. A. and Wilson, M. L., “Admixtures use in concrete,” *CD039, PCA, Skokie, IL,* 2002.

[30] Flatt, R. J. and Houst, Y. F., “A simplified view on chemical effects perturbing the action of superplasticizers,” *Cement and Concrete Research,* 2001, 31, pp 1169-1176.

[31] “Standard test method for microscopical determination of parameters of the air-void system in hardened concrete,” ASTM C-457, 2009.

[32] Sovannsathya, R., Anuwat, A., and Ouchi, M. “An effective and efficient mixing method for controlling initial air content with stability of entrained air in fresh mortar of self-compacting concrete, ”*Proceedings of the Japan Concrete Institute,* 2015, 37(1), pp 1447-1452.

[33] Sovannsathya, R., Anuwat, A. and Ouchi, M., “Fineness of air bubbles affected by mixing procedure in mortar in self-compacting concrete,” Proceedings of JCI, Vol. 38(1), 2016, pp.

[34] Sovannsathya, R., Anuwat, A. and Ouchi, M., “An effective and efficient mixing method for controlling initial air content with stability of entrained air in fresh mortar of self-compacting concrete,” *Internet Journal for Society of Management System,* SMS15-6550, 2016

[35] Anuwat, A., Tanaka, K., Sovannsathya, R., and Ouchi, M., “Air-enhanced self-compactability of fresh concrete with effective mixing method, ”*Proceedings of the Japan Concrete Institute,* 2015, 37, pp.

# APPENDIX A: AIR ENTRAINMENT CHARACTERISTICS MEASURED WITH LINEAR TRAVERSE METHOD

**Table A.1 Fresh mortar properties made by different mix-proportion and mixing procedure, each data was the result mean value from 3 mortar samples test**

| | | Test just after mixing | | | Test at 2 hours after the first contact of cement and water | | |
|---|---|---|---|---|---|---|---|
| N° | Mix-proportion and mixing procedure | Air (%) | Flow (mm) | $R_m$ | $A_{2h}$ (%) | $Flow_{2h}$ (mm) | $R_{m,2h}$ |
| $1_{m*}$ | $SP_1$(1.4%)-$AE_2$(0.04%)-A | 18.0 | 259.8 | 2.25 | 14.7 | 244.0 | 1.64 |
| $2_m$ | $SP_1$(1.4%)-$AE_2$(0.20%)-A | 29.7 | 178.0 | 1.56 | 24.8 | 169.5 | 0.96 |
| $3_m$ | $SP_1$(1.4%)-$AE_2$(0.04%)-B | 7.0 | 270.0 | 3.19 | 8.4 | 233.0 | 1.95 |
| $4_m$ | $SP_1$(1.4%)-$AE_2$(0.20%)-B | 8.9 | 255.0 | 3.08 | 10.8 | 200.7 | 1.63 |
| $5_m$ | $SP_1$(1.4%)-$AE_2$(0.04%)-C | 10.0 | 268.0 | 2.36 | 10.3 | 231.8 | 1.47 |
| $6_m$ | $SP_1$(1.4%)-$AE_2$(0.20%)-C | 11.5 | 262.5 | 2.45 | 13.3 | 220.2 | 1.49 |
| $7_m$ | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,1}$ | 13.6 | 235.0 | 2.23 | 14.3 | 233.2 | 1.53 |
| $8_m$ | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,2}$ | 17.7 | 239.7 | 2.32 | 17.9 | 235.5 | 1.65 |
| $9_m$ | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,3}$ | 20.5 | 254.5 | 2.38 | 20.1 | 237.5 | 1.66 |
| $10_m$ | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.2 | 256.3 | 2.65 | 16.0 | 262.3 | 1.87 |
| $11_m$ | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0,2}$ | 25.0 | 170.0 | 1.40 | 19.6 | 239.0 | 1.36 |
| $12_m$ | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0.5,2}$ | 19.7 | 222.5 | 1.93 | 18.6 | 264.0 | 1.77 |
| $13_m$ | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{1,2}$ | 16.3 | 254.0 | 2.42 | 16.2 | 266.2 | 2.05 |
| $14_m$ | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{2,2}$ | 17.1 | 260.3 | 2.57 | 17.0 | 266.3 | 2.04 |
| $15_m$ | $SP_2$(0.5%)-$AE_1$(0.015%)-$C_{2,2}$ | 18.4 | 181.7 | 2.29 | 18.3 | 100.0 | 0.74 |
| $16_m$ | $SP_2$(0.7%)-$AE_1$(0.015%)-$C_{2,2}$ | 17.7 | 243.3 | 2.79 | 17.9 | 219.2 | 1.95 |
| $17_m$ | $SP_2$(0.8%)-$AE_1$(0.015%)-$C_{2,2}$ | 16.5 | 263.0 | 3.10 | 16.7 | 253.2 | 2.21 |
| $18_m$ | $SP_1$(1.3%)-$AE_1$(0.015%)-$C_{1,3}$ | 15.6 | 268.0 | 2.97 | 15.7 | 249.0 | 1.96 |
| $19_m$ | $SP_1$(1.4%)-$AE_2$(0.06%)-$C_{2,2}$ | 15.4 | 263.0 | 2.62 | 15.5 | 252.5 | 1.86 |
| $20_m$ | $SP_2$(0.8%)-$AE_1$(0.013%)-$C_{2,1.75}$ | 13.6 | 265.0 | 3.22 | 13.5 | 262.0 | 2.13 |
| $21_m$ | $SP_2$(1.0%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.6 | 266.5 | 2.32 | 15.7 | 268.5 | 1.92 |

** m: mean value from 3 mortar samples test*

**Table A.2 Fresh mortar properties made by different mix-proportion and mixing procedure, mortar samples for air-voids distribution test**

| | | Test just after mixing | | | Test at 2 hours after the first contact of cement and water | | |
|---|---|---|---|---|---|---|---|
| N° | Mix-proportion and mixing procedure | Air (%) | Flow (mm) | $R_m$ | $A_{2h}$ (%) | $Flow_{2h}$ (mm) | $R_{m,2h}$ |
| 1 | $SP_1$(1.4%)-$AE_2$(0.04%)-A | 17.5 | 259.0 | 2.35 | 14.4 | 240.5 | 1.62 |
| 2 | $SP_1$(1.4%)-$AE_2$(0.20%)-A | 29.4 | 180.5 | 1.56 | 24.6 | 171.0 | 0.96 |
| 3 | $SP_1$(1.4%)-$AE_2$(0.04%)-B | 7.0 | 270.0 | 3.27 | 8.1 | 234.5 | 2.04 |
| 4 | $SP_1$(1.4%)-$AE_2$(0.20%)-B | 8.9 | 258.5 | 3.18 | 10.6 | 199.0 | 1.71 |
| 5 | $SP_1$(1.4%)-$AE_2$(0.04%)-C | 9.8 | 267.5 | 2.26 | 10.4 | 234.0 | 1.40 |
| 6 | $SP_1$(1.4%)-$AE_2$(0.20%)-C | 11.3 | 264.5 | 2.46 | 13.1 | 219.0 | 1.54 |
| 7 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,1}$ | 13.1 | 235.0 | 2.35 | 14.0 | 231.5 | 1.60 |
| 8 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,2}$ | 17.2 | 238.5 | 2.39 | 17.4 | 232.5 | 1.59 |
| 9 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,3}$ | 21.0 | 249.5 | 2.27 | 20.4 | 237.0 | 1.69 |
| 10 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.3 | 260.5 | 2.66 | 16.0 | 265.5 | 1.88 |
| 11 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0,2}$ | 27.9 | 156.0 | 1.32 | 21.4 | 240.0 | 1.24 |
| 12 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0.5,2}$ | 19.0 | 233.0 | 2.02 | 18.3 | 257.0 | 1.80 |
| 13 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{1,2}$ | 16.2 | 255.0 | 2.50 | 16.0 | 263.0 | 2.06 |
| 14 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{2,2}$ | 17.3 | 261.0 | 2.62 | 17.3 | 266.5 | 2.10 |
| 15 | $SP_2$(0.5%)-$AE_1$(0.015%)-$C_{2,2}$ | 18.3 | 184.0 | 2.31 | 17.9 | 100.0 | 0.58 |
| 16 | $SP_2$(0.7%)-$AE_1$(0.015%)-$C_{2,2}$ | 17.9 | 244.5 | 2.82 | 18.1 | 222.5 | 1.98 |
| 17 | $SP_2$(0.8%)-$AE_1$(0.015%)-$C_{2,2}$ | 17.0 | 260.5 | 2.98 | 17.2 | 248.0 | 2.11 |
| 18 | $SP_1$(1.3%)-$AE_1$(0.015%)-$C_{1,3}$ | 15.2 | 264.0 | 3.09 | 15.1 | 261.0 | 2.02 |
| 19 | $SP_1$(1.4%)-$AE_2$(0.06%)-$C_{2,2}$ | 15.1 | 264.5 | 2.63 | 15.5 | 248.0 | 1.82 |
| 20 | $SP_2$(0.8%)-$AE_1$(0.013%)-$C_{2,1.75}$ | 15.0 | 265.5 | 3.10 | 14.9 | 257.0 | 2.25 |
| 21 | $SP_2$(1.0%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.3 | 268.0 | 2.83 | 15.0 | 267.5 | 2.14 |

**Table A.3 Air-void distribution properties of mortar made by different mix-proportion and mixing procedure**

| | | At fresh state | | At hardened state measure by LTM | | |
|---|---|---|---|---|---|---|
| N° | Mix-proportion and mixing procedure | Air (%) | $A_{2h}$ (%) | $A_{har.}$ (%) | α* ($mm^2/mm^3$) | ($A_{har.}$ × α)** |
| 1 | $SP_1$(1.4%)-$AE_2$(0.04%)-A | 17.5 | 14.4 | 12.8 | 14.79 | 188.8 |
| 2 | $SP_1$(1.4%)-$AE_2$(0.20%)-A | 29.4 | 24.6 | 22.0 | 15.69 | 345.3 |
| 3 | $SP_1$(1.4%)-$AE_2$(0.04%)-B | 7.0 | 8.1 | 7.1 | 17.35 | 123.8 |
| 4 | $SP_1$(1.4%)-$AE_2$(0.20%)-B | 8.9 | 10.6 | 8.8 | 22.38 | 196.2 |
| 5 | $SP_1$(1.4%)-$AE_2$(0.04%)-C | 9.8 | 10.4 | 10.6 | 14.65 | 154.8 |
| 6 | $SP_1$(1.4%)-$AE_2$(0.20%)-C | 11.3 | 13.1 | 12.0 | 18.02 | 216.8 |
| 7 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,1}$ | 13.1 | 14.0 | 14.3 | 15.03 | 215.0 |
| 8 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,2}$ | 17.2 | 17.4 | 17.4 | 17.17 | 299.3 |
| 9 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{1,3}$ | 21.0 | 20.4 | 17.3 | 16.53 | 285.5 |
| 10 | $SP_1$(1.4%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.3 | 16.0 | 15.6 | 17.12 | 267.7 |
| 11 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0,2}$ | 27.9 | 21.4 | 18.8 | 14.97 | 281.0 |
| 12 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{0.5,2}$ | 19.0 | 18.3 | 15.3 | 16.15 | 247.7 |
| 13 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{1,2}$ | 16.2 | 16.0 | 14.3 | 16.48 | 236.3 |
| 14 | $SP_2$(0.9%)-$AE_2$(0.08%)-$C_{2,2}$ | 17.3 | 17.3 | 16.2 | 16.49 | 266.6 |
| 15 | $SP_2$(0.5%)-$AE_1$(0.015%)-$C_{2,2}$ | 18.3 | 17.9 | 17.0 | 18.13 | 308.8 |
| 16 | $SP_2$(0.7%)-$AE_1$(0.015%)-$C_{2,2}$ | 17.9 | 18.1 | 17.3 | 20.74 | 358.1 |
| 17 | $SP_2$(0.8%)-$AE_1$(0.015%)-$C_{2,2}$ | 17.0 | 17.2 | 15.9 | 20.89 | 331.5 |
| 18 | $SP_1$(1.3%)-$AE_1$(0.015%)-$C_{1,3}$ | 15.2 | 15.1 | 13.9 | 17.75 | 246.1 |
| 19 | $SP_1$(1.4%)-$AE_2$(0.06%)-$C_{2,2}$ | 15.1 | 15.5 | 15.5 | 17.40 | 269.1 |
| 20 | $SP_2$(0.8%)-$AE_1$(0.013%)-$C_{2,1.75}$ | 15.0 | 14.9 | 13.9 | 18.78 | 261.7 |
| 21 | $SP_2$(1.0%)-$AE_2$(0.08%)-$C_{2,2}$ | 15.3 | 15.0 | 13.5 | 18.03 | 243.4 |

* α: specific surface of air

** Total surface area of air ($mm^2$/100 $mm^3$ of mortar)

# APPENDIX B: AIR ENTRAINMENT CHARACTERISTICS MEASURED WITH AIR-VOID ANALYZER (AVA-3000)

**Note:** The air entrainment characteristics presented here are the raw data obtained with AVA-3000 test results. There is no adjustment on the air loss during sampling the mortar sample for air size distribution test.

**Sample N° 1: $A_2$-AE0.005%-SP1.4%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 7.6 | 6.4 | 3.0 |
| Air in paste (%) | 16.3 | 13.5 | 6.5 |
| Specific surface ($mm^2/mm^3$) | 13.7 | 15.5 | |
| Spacing factor (mm) | 0.364 | 0.348 | |

**Sample N° 2: $A_6$-AE0.005%-SP1.2%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 5.9 | 4.9 | 2.3 |
| Air in paste (%) | 12.1 | 10.0 | 4.8 |
| Specific surface ($mm^2/mm^3$) | 14.3 | 16.2 | |
| Spacing factor (mm) | 0.393 | 0.375 | |

**Sample N° 3: $A_2$-AE0.010%-SP1.4%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 12.8 | 11.9 | 7.2 |
| Air in paste (%) | 28.5 | 26.2 | 15.9 |
| Specific surface ($mm^2/mm^3$) | 18.2 | 19.4 | |
| Spacing factor (mm) | 0.193 | 0.194 | |

**Sample N° 4: $A_4$-AE0.010%-SP1.4%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 18.1 | 15.9 | 8.4 |
| Air in paste (%) | 40.1 | 34.4 | 18.7 |
| Specific surface ($mm^2/mm^3$) | 17.0 | 19.1 | |
| Spacing factor (mm) | 0.147 | 0.149 | |

**Sample N° 5: $A_6$-AE0.010%-SP1.2%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 19.4 | 16.3 | 7.7 |
| Air in paste (%) | 41.7 | 33.8 | 16.6 |
| Specific surface ($mm^2/mm^3$) | 15.6 | 18.3 | |
| Spacing factor (mm) | 0.148 | 0.151 | |

**Sample N° 6: $A_2$-AE0.020%-SP1.4%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 19.7 | 19.6 | 14.5 |
| Air in paste (%) | 44.6 | 44.2 | 32.8 |
| Specific surface ($mm^2/mm^3$) | 22.1 | 22.3 | |
| Spacing factor (mm) | 0.103 | 0.103 | |

**Sample N° 7: $A_4$-AE0.020%-SP1.2%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 27.4 | 26.8 | 18.9 |
| Air in paste (%) | 64.6 | 62.8 | 44.6 |
| Specific surface ($mm^2/mm^3$) | 19.9 | 20.4 | |
| Spacing factor (mm) | 0.082 | 0.082 | |

**Sample N° 8: $A_6$-AE0.020%-SP1.2%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 28.8 | 28.5 | 21.1 |
| Air in paste (%) | 68.4 | 67.3 | 49.9 |
| Specific surface ($mm^2/mm^3$) | 21.8 | 22.1 | |
| Spacing factor (mm) | 0.072 | 0.071 | |

**Sample N° 9: $A_8$-AE0.020%-SP1.2%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 20.3 | 20.1 | 15.2 |
| Air in paste (%) | 45.2 | 44.5 | 33.8 |
| Specific surface ($mm^2/mm^3$) | 22.0 | 22.2 | |
| Spacing factor (mm) | 0.101 | 0.101 | |

**Sample N° 10: $A_2$-AE0.005%-SP0.7%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 9.6 | 7.1 | 2.2 |
| Air in paste (%) | 20.8 | 15.0 | 4.9 |
| Specific surface ($mm^2/mm^3$) | 9.0 | 10.9 | |
| Spacing factor (mm) | 0.500 | 0.476 | |

**Sample N° 11: $A_2$-AE0.010%-SP0.7%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 10.5 | 8.3 | 4.1 |
| Air in paste (%) | 21.8 | 16.8 | 8.6 |
| Specific surface ($mm^2/mm^3$) | 14.5 | 17.6 | |
| Spacing factor (mm) | 0.297 | 0.273 | |

**Sample N° 12: $B_{1,2}$-AE0.010%-SP1.1%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 5.6 | 5.2 | 3.0 |
| Air in paste (%) | 12.3 | 11.3 | 6.5 |
| Specific surface ($mm^2/mm^3$) | 14.4 | 15.2 | |
| Spacing factor (mm) | 0.400 | 0.391 | |

**Sample N° 13: $B_{1,4}$-AE0.010%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 6.9 | 6.5 | 4.6 |
| Air in paste (%) | 15.1 | 14.1 | 10.1 |
| Specific surface ($mm^2/mm^3$) | 19.7 | 20.8 | |
| Spacing factor (mm) | 0.266 | 0.259 | |

**Sample N° 14: $B_{1,6}$-AE0.010%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 7.1 | 6.8 | 4.9 |
| Air in paste (%) | 15.8 | 15.0 | 10.9 |
| Specific surface ($mm^2/mm^3$) | 19.9 | 20.8 | |
| Spacing factor (mm) | 0.259 | 0.253 | |

**Sample N° 15: $B_{1,10}$-AE0.015%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 8.6 | 8.3 | 6.4 |
| Air in paste (%) | 19.1 | 18.4 | 14.4 |
| Specific surface ($mm^2/mm^3$) | 23.2 | 23.9 | |
| Spacing factor (mm) | 0.204 | 0.201 | |

**Sample N° 16: $B_{1,15}$-AE0.015%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 12.0 | 11.6 | 9.5 |
| Air in paste (%) | 26.9 | 25.8 | 21.2 |
| Specific surface ($mm^2/mm^3$) | 25.9 | 26.9 | |
| Spacing factor (mm) | 0.144 | 0.145 | |

**Sample N° 17: $B_{1,20}$-AE0.015%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 15.6 | 15.8 | 13.3 |
| Air in paste (%) | 35.0 | 35.4 | 29.8 |
| Specific surface ($mm^2/mm^3$) | 26.6 | 26.4 | |
| Spacing factor (mm) | 0.108 | 0.108 | |

**Sample N° 18: $B_{1,2}$-AE0.020%-SP1.1%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 6.7 | 6.3 | 4.5 |
| Air in paste (%) | 15.0 | 14.1 | 10.0 |
| Specific surface ($mm^2/mm^3$) | 19.8 | 20.8 | |
| Spacing factor (mm) | 0.267 | 0.261 | |

**Sample N° 19: $B_{1,4}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 7.8 | 7.8 | 6.6 |
| Air in paste (%) | 17.3 | 17.1 | 14.5 |
| Specific surface ($mm^2/mm^3$) | 26.0 | 26.2 | |
| Spacing factor (mm) | 0.190 | 0.189 | |

**Sample N° 20: $B_{1,6}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 9.4 | 9.1 | 7.7 |
| Air in paste (%) | 21.2 | 20.3 | 17.2 |
| Specific surface ($mm^2/mm^3$) | 27.3 | 28.3 | |
| Spacing factor (mm) | 0.166 | 0.163 | |

**Sample N° 21: $B_{1,10}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 10.8 | 10.5 | 8.5 |
| Air in paste (%) | 24.1 | 23.2 | 18.8 |
| Specific surface ($mm^2/mm^3$) | 25.1 | 25.9 | |
| Spacing factor (mm) | 0.166 | 0.166 | |

**Sample N° 22: $B_{1,15}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 12.8 | 12.3 | 10.5 |
| Air in paste (%) | 28.7 | 27.5 | 23.6 |
| Specific surface ($mm^2/mm^3$) | 27.9 | 29.0 | |
| Spacing factor (mm) | 0.126 | 0.126 | |

**Sample N° 23: $B_{1,20}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 20.9 | 20.8 | 17.3 |
| Air in paste (%) | 50.1 | 49.6 | 41.3 |
| Specific surface ($mm^2/mm^3$) | 26.6 | 26.8 | |
| Spacing factor (mm) | 0.081 | 0.081 | |

**Sample N° 24: $B_{1,25}$-AE0.020%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 19.6 | 19.4 | 16.9 |
| Air in paste (%) | 43.5 | 43.1 | 37.5 |
| Specific surface ($mm^2/mm^3$) | 28.6 | 28.8 | |
| Spacing factor (mm) | 0.081 | 0.081 | |

**Sample N° 25: $B_{1,4}$-AE0.040%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 11.1 | 10.0 | 7.6 |
| Air in paste (%) | 25.0 | 22.4 | 17.1 |
| Specific surface ($mm^2/mm^3$) | 20.6 | 22.7 | |
| Spacing factor (mm) | 0.197 | 0.194 | |

**Sample N° 26: $B_{1,6}$-AE0.040%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 11.7 | 11.3 | 9.9 |
| Air in paste (%) | 26.0 | 25.2 | 22.1 |
| Specific surface ($mm^2/mm^3$) | 31.6 | 32.4 | |
| Spacing factor (mm) | 0.122 | 0.122 | |

**Sample N° 27: $B_{1,8}$-AE0.040%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 13.3 | 13.0 | 10.8 |
| Air in paste (%) | 29.8 | 29.1 | 24.2 |
| Specific surface ($mm^2/mm^3$) | 25.3 | 25.8 | |
| Spacing factor (mm) | 0.134 | 0.134 | |

**Sample N° 28: $B_{1,6}$-AE0.080%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 13.1 | 12.8 | 11.0 |
| Air in paste (%) | 29.0 | 28.1 | 24.3 |
| Specific surface ($mm^2/mm^3$) | 26.8 | 27.4 | |
| Spacing factor (mm) | 0.128 | 0.129 | |

**Sample N° 29: $B_{1,8}$-AE0.080%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 14.1 | 13.9 | 13.3 |
| Air in paste (%) | 31.2 | 30.8 | 29.5 |
| Specific surface ($mm^2/mm^3$) | 36.0 | 36.5 | |
| Spacing factor (mm) | 0.089 | 0.089 | |

**Sample N° 30: $B_{1,10}$-AE0.080%-SP0.9%**

| Chord length | <2 mm | <1 mm | <0.35 mm |
|---|---|---|---|
| Air in mortar (%) | 17.1 | 16.7 | 14.8 |
| Air in paste (%) | 38.6 | 37.6 | 33.4 |
| Specific surface ($mm^2/mm^3$) | 29.3 | 30.1 | |
| Spacing factor (mm) | 0.090 | 0.090 | |